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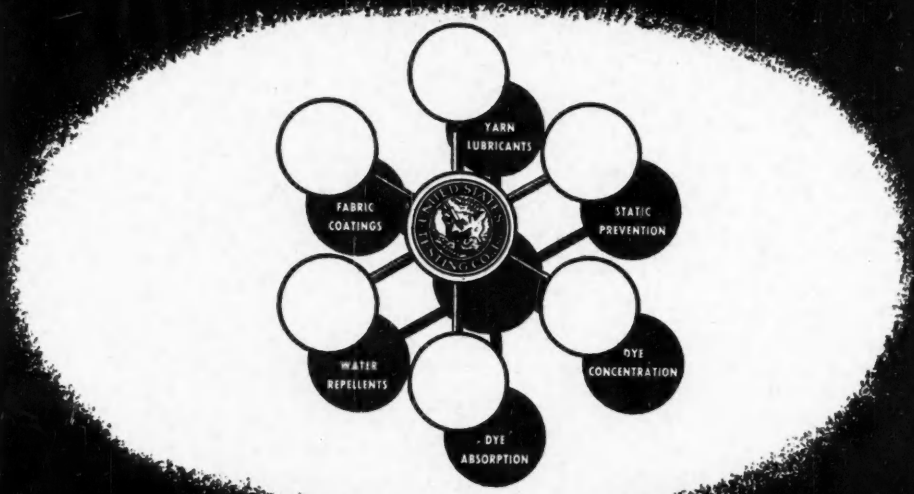

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TEXTILE RESEARCH JOURNAL

VOL. XIX, NO. 8 AUGUST, 1949

Shrinkage Control of Wool by Melamine Resins

Part I: Microscopical Observations

C. Maresh and G. L. Royer

American Cyanamid Company, Calco Chemical Division, Bound Brook, New Jersey

THE FELTING AND SHRINKING of wool is generally considered to be due to the rootward movement of the individual scale-surfaced fibers within individual threads [1, 18, 19], leading eventually to a compact tangled mass [5*a*]. The cause of migration is thought to be due primarily to the frictional differences [15, 17] which exist between the tip-to-root and root-to-tip directions of the fiber. This can be easily demonstrated by rubbing a wool fiber between the thumb and forefinger. The fiber will travel in the root direction.

The extremely elastic cortex capable of swelling in water and the much less elastic and less hydrophylic scale structure, together with surface frictional differences, are probably the major fiber properties which cause shrinkage. External mechanical motion on the fibers with such properties finally leads to a compact mass in which further movement of fibers is severely restricted.

If it is assumed that the above concepts are correct, two methods become apparent for attaining felting and shrinkage control:

1. Removal or alteration of portions of the epidermis, particularly the scale overlap, by either chemical or physical means.

2. Addition to the fiber of a material which will penetrate into, adhere to, or react with the various components of the wool fibers, thereby altering their frictional and plastic behavior.

The first method is exemplified by many chemical processes being used today to stabilize wool. Some typical chemical treatments are permanganate, sodium sulfide, chlorine in acid and alkaline solution, sulfuryl chloride, sulfite and peroxide, hypochlorite with formic acid, sodium salts of phosphoric acid and formaldehyde, and alcoholic KOH. Enzymes such as trypsin probably also react in this manner.

According to Speakman [20], it is possible to make wool unshrinkable by mechanical degradation with abrasives. The working of wool with certain pigments has been found to produce shrinkage control.

There is evidence that the pigment particles become lodged at scale interstices, thus altering the

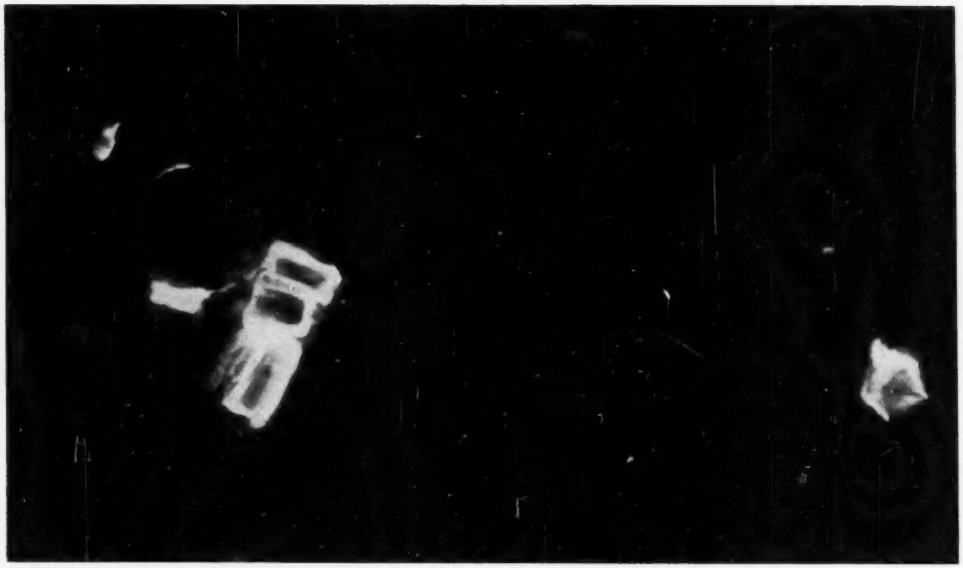

FIG. 1. *Cross section of* LANASET*-treated wool fiber photographed through phase microscope. Magnification 1000 ×.*

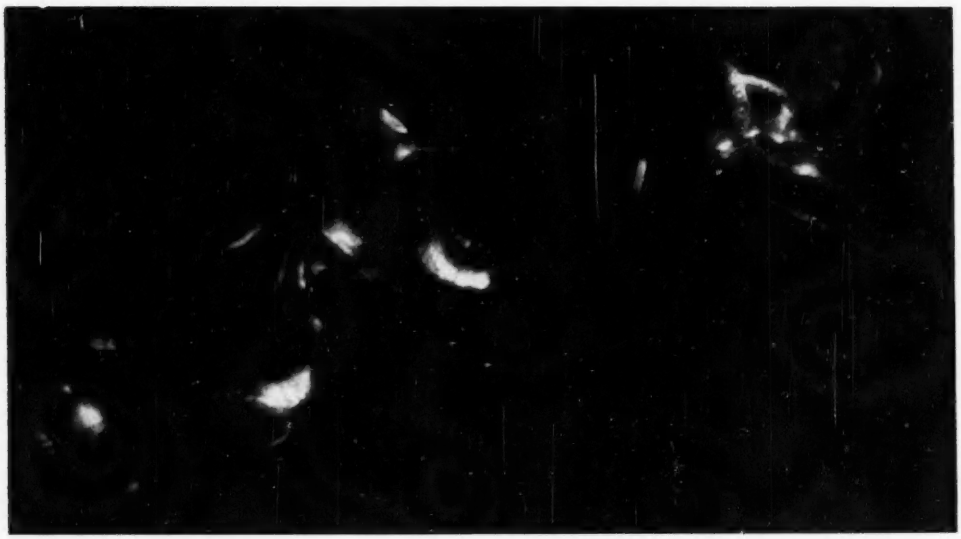

FIG. 2. LANASET*-treated wool fiber after removal of cortex with sodium sulfide solution, leaving sausage-casing effect of resin-bonded or strengthened scale structure. Photographed through phase microscope. Magnification 1000 ×.*

FIG. 3. LANASET RESIN-*treated wool fiber showing deposit essentially at scale interstices. Photographed through phase microscope. Magnification 1000 ×.*

FIG. 4. *Untreated wool fiber. Photographed through phase microscope. Magnification 1000 ×.*

frictional properties or restricting the movement of the scale with respect to the cortex. Possibly abrasive materials act by this mechanism rather than by removal of scales.

The second method for attaining shrinkage control of wool is exemplified by linkage formation within the fibers as proposed by Speakman *et al.* [19] or by the use of thermosetting resins [4] such as LANASET * RESIN. The mechanism of methylated methylol melamine resin in controlling shrinkage is thought to depend to a great extent upon its location in or on the fiber.

Theoretically, the wool fiber could be made nonshrinkable by permeating the intercellular substance of the wool fiber with resin. F. T. Marshall and S. J. Aulabaugh [13] have reported the existence of melamine resin in the cortex of the wool fiber by a staining technique. It was reported by one of the present authors [6] that observations of dyeing made with an acid-type dye on treated and untreated fibers indicated the presence of resin within the wool fiber itself. However, it has been subsequently found that treatment of a wool fiber with formaldehyde alone produces the same degree of resistance to penetration of color into freshly cut fiber ends as the melamine resin does. Sufficient free formaldehyde is always present in melamine resins to account for this behavior. Similarly, Landolt [7] has shown that the swelling of cross-sectional slips of melamine-formaldehyde resin-treated wool fiber in caustic is much less than that of untreated fiber. Formaldehyde is known to reduce the swelling of wool fiber in water solutions. Norman *et al.* [16] have obtained information from emission-band spectroscopy that melamine-formaldehyde resin reacts with the wool. There is no indication from their work as to where the reaction takes place—that is, whether it is in the epidermis or the cortex portions of the fiber. Stock and Salley [21] showed that tensile hysteresis cycles for wet fibers before and after resin treatment are equal under conditions of stress-strain equilibrium. This has been substantiated by the work of McCleary and Royer [14] (Table II, page 460). On the basis of these latter results, it is doubtful whether any resin solution penetrates very far into the fiber. In this laboratory direct staining of resin in the cortex by the method of Marshall and Aulabaugh [13] was not confirmed. Phase-microscopical examination of thin cross sections of treated fibers showed no evidence of the presence of resin in the cortex. However, Figure 1 does show spots of resin just beneath the scale.

One explanation for the mechanism of shrinkage control by methylated methylol melamine resin is that it alters the scale characteristics, and/or the physical relationship between the scale and the cortex portions of the fiber. Lindberg, Philips, and Gralén [11] have shown the occurrence of a thin sodium sulfide-insoluble membrane appearing in or on the surface structure of the scale portions of the wool fiber. E. Lehmann [8] has, furthermore, shown the existence of a histological structure which he calls the "epidermis membrane" under the scale layer of animal fibers when the fiber has been pretreated with formaldehyde and subsequently treated with alkali. Figure 2 shows a wool fiber which has been treated with methylated methylol melamine resin and subsequently digested with sodium sulfide for 10 days. A thin sausage-like casing of membrane remains, apparently held together by the resin. The relationship of this membrane, cemented by resin in the form of a sausage casing around the fiber, to either the membranes of Lehmann or Lindberg *et al.* is not clear at this point; however, it does indicate the location of the methylated methylol melamine resin as being essentially in the scale portions of the fiber.

Further discussion of the membrane by Lindberg [9] indicates that the membrane appears on the surface of the scale and is very likely responsible for the Allwörden's sacs. The skin is said to be about 100 A. thick and each sac seems to cover one scale, but sometimes a large sac covers several scales. The extreme sensitivity of Whewell and Wood's [22] test for mechanical damage, as well as the Kiton Red G test [3] for determining extent of chemical damage, can be explained by this membrane. According to the experiments of Barker [2], the long-standing supposition that there are scales on wool is incorrect and the theory that there is a sheath formation is more correct.

It is well known that the physical condition of the wool will influence the pick-up of dyes and, similarly, the pick-up of resin. Figure 3 shows a fiber treated with a methylated methylol melamine resin and illustrates the distribution of the resin on the fiber surface. This fiber was mounted in an immersion liquid of refractive index 1.516. The wool fiber would normally be practically invisible except

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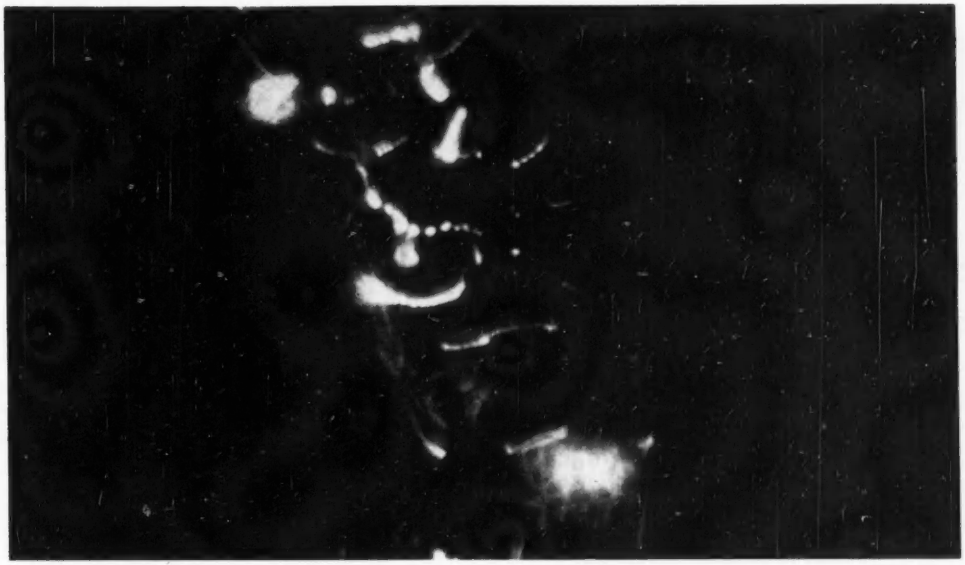

FIG. 5. LANASET-*treated wool fiber during extraction with sodium sulfide. Swelling has caused splitting of resin at scale interstices. Photographed through phase microscope. Magnification 1000 ×.*

for the resin located at the scale interstices. Figure 4 shows an untreated fiber similarly mounted. Both photomicrographs were taken with the phase microscope. The photomicrographs indicate that the resin becomes anchored at the scale interstices. The membrane covering is most likely to be opened up at the scale interstices, and it is at these points that mechanical or chemical action first becomes evident when the specimen is stained according to the methods of Whewell and Wood [22] and Carter and Consden [3].

Good shrinkage control is attainable when sufficient resin is deposited on the fiber as shown by Figure 3. This photomicrograph represents an ideal distribution of resin which is usually not obtained commercially.

The recently announced method of application of LANASET RESIN [5] whereby the wool is made more receptive to the resin by a peroxide pretreatment results in better distribution of the resin on the fiber. The peroxide pretreatment causes the opening up of the fiber at the scale interstices so that there is better anchorage of the resin at these points.

Comparison of the disintegration of untreated and methylated methylol melamine resin-treated wool fiber in a 0.1*N* sodium sulfide solution shows striking differences. The untreated fiber shows swelling of the fiber followed by peeling of the scale structure and disintegration of the cortex into fibrils. After approximately 30 days, the fibrils are completely digested and the solution contains the colloidally dispersed scale membrane. The methylated methylol melamine resin-treated fibers are considerably more resistant to sodium sulfide digestion. Except where the scale interstices are not filled in with resin, the dissolution of the fibers takes place through the cut ends of the fibers. Some swelling is indicated by breaks appearing in the resin deposits at the scale interstices, as shown in Figure 5.

If it is assumed that the resin is located essentially at the scale interstices, what, then, is the mechanism of the shrinkage control so obtained? It has been reported by Lindberg and Gralén [10] as well as by Makinson [12] that methylated methylol melamine resin does not materially reduce the frictional differences. Stock and Salley [21], however, report a significant decrease in the directional coefficient and increased friction in both directions as a consequence of the treatment of individual fibers. The difference is questionable for fibers drawn

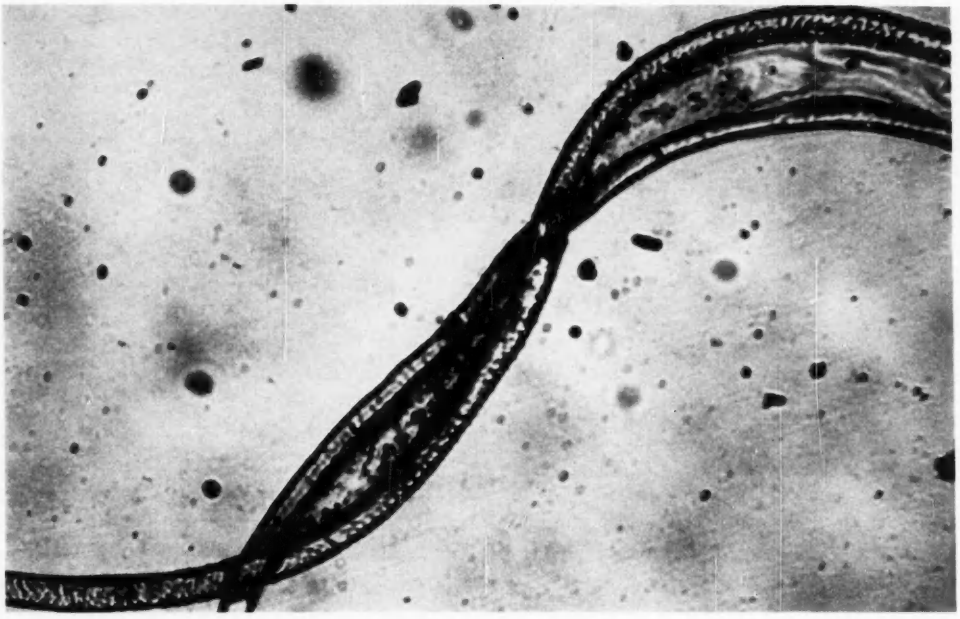

FIG. 6. *Two wool fibers bonded together with* LANASET RESIN. *Magnification 200 ×.*

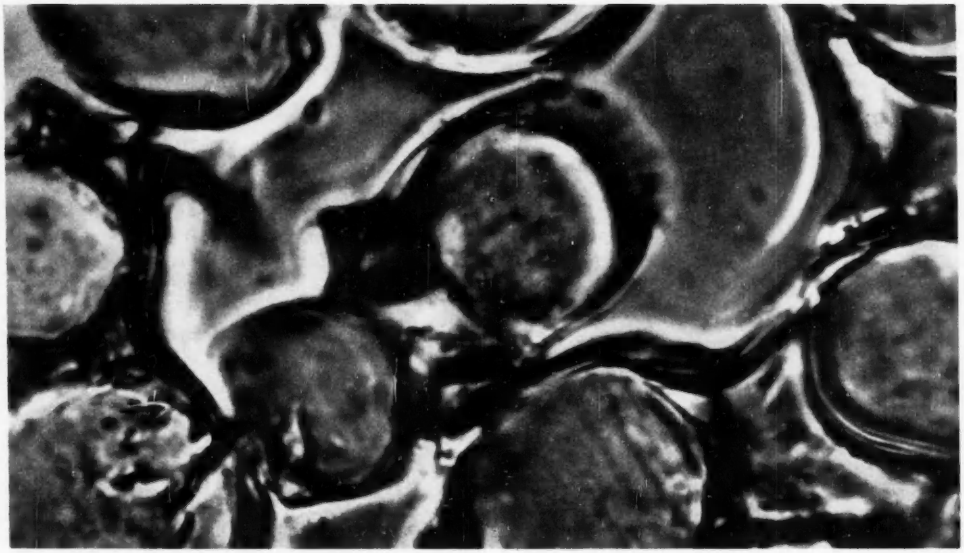

FIG. 7. *Cross section of wool fibers from* LANASET RESIN-*treated fabric showing resin bridging or bonding of fibers. Magnification 1000 ×.*

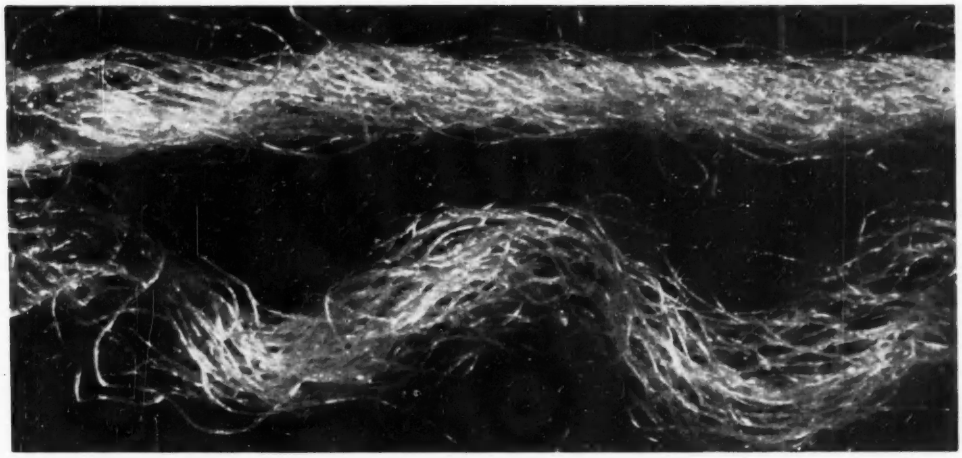

FIG. 8. Top—*Thread removed from untreated wool fabric.* Bottom—*Thread removed from* LANASET RESIN-*treated fabric showing stabilization of entire thread. Magnification 20 ×.*

from treated and untreated lengths of slivers of loose parallel fibers and there is no difference for fibers withdrawn from threads raveled from treated and untreated cloth. They explain that the failure to find a change in friction for fibers withdrawn from cloths may be due to (1) resin solution not being deposited on fibers in mass as effectively as on single fibers or (2) removal of fibers from the mass, causing much of the resin to be broken loose. It is further known that the fibers cannot be treated successfully in the raw or sliver state and give satisfactory control in the finished goods. This might then indicate that the effects of the resin upon the frictional properties of the epidermis are only one

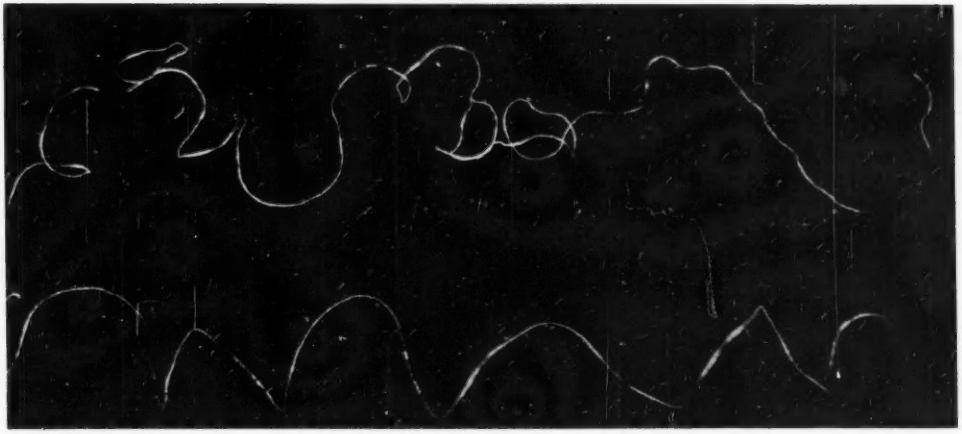

FIG. 9. Bottom—LANASET RESIN-*treated wool fiber cured in form of coil; note stabilization.* Top—*Untreated wool fiber subjected to same treatment except without the* LANASET RESIN *shows complete lack of stabilization. Magnification 20 ×.*

factor and that an additional mechanism in resin shrinkage control is reduction of the physical differences between the epidermis and the cortex either by making the epidermis more continuous or by providing bonding between the scale and the cortex at the scale interstices. It has been observed microscopically that the resin does not flow out evenly over the entire fiber surface during the drying and curing period. The resin tends to collect at the scale interstices and at points of contact between fibers. It must be assumed that local spot-welding of one fiber to another does play some part in the shrinkage control of wool. Figures 6 and 7 represent longitudinal and cross-sectional views of fibers removed from melamine-resin laboratory-treated wool fabrics. Figure 6 shows two fibers cemented together. Figure 7 shows four fibers held together rigidly by resin. It is believed that excessive spot-welding as illustrated by these photographs makes for poor "hand." The recent developments of LANASET RESIN application [5] reduce to a minimum such spot-welding and thereby eliminate loss of "hand" due to this factor. It is also thought that impregnation of the cortex with this type of resin (it has been indicated earlier that no proof of such penetration has been obtained) would likewise cause stiffness. The location of the resin essentially at the vulnerable scale interstices, as is the case when LANASET * RESIN is applied under recommended procedures, would produce control with the least sacrifice of hand. Over-all control of threads and fibers is shown by Figures 8 and 9, respectively. In Figure 9 the single untreated and treated fibers were wound around small glass rods. Treatment of the lower fiber with methylol melamine resin followed by curing caused it to become set in the position in which it was held during the drying and curing period so that it kept this form even when placed in water. The upper fiber was immersed in water and heated in the oven at the same resin curing temperature. Upon removal from the rod, the fiber relaxed to its original state when placed in water. This indicates that control is obtained in a single fiber by factors other than fiber-bonding or so-called spot-welding.

Summary

Microscopical examination of methylated methylol melamine resin-treated wool fibers indicates that the resin is located essentially at the scale region of the fiber. The mechanism by which the modification of this portion of the fiber produces shrinkage control is at least twofold: (1) the change in the directional coefficient caused by the deposition of the resin essentially at the scale interstices and (2) the change in the elastic properties of the fiber or the so-called "setting" of the fiber, not by impregnation thereof, but rather by the formation of a semi-rigid sheath around the elastic cortex. The role played by fiber-bonding is not to be discounted, for it undoubtedly plays some part in the control of shrinkage in wool treated with methylated melamine-formaldehyde resin.

Acknowledgment

The authors would like to acknowledge the assistance of, and discussion with, the other members of the Microscopical Laboratory, particularly Mr. R. R. Krammes and Miss F. Gustafson, as well as the guidance and assistance of Dr. R. H. Kienle, under whose direction this work was carried out.

Literature Cited

1. Arnold, H., *Leipzig. Monats. Textilind.* **44**, 463, 507, 540 (1929).
2. Barker, A. F., *Textile Manufacturer* **1948**, 256–8; 313–15.
3. Carter, E. G. H., and Consden, R., *J. Text. Inst.* **37**, T227 (1946).
4. Dudley, J. R., and Lynn, J. E., Soc. Dyers and Colourists, "Symposium on Fibrous Proteins," 1946, p. 215.
5. Fluck, L. A., Report presented at National Assoc. Wool Mfgs. 84th Annual Meeting, Waldorf-Astoria Hotel, New York, April 14, 1949.
5a. Harris, Milton, *Am. Dyestuff Reptr.* **34**, 72 (1945).
6. Kienle, R. H., Royer, G. L., and McCleary, H. R., *Am. Dyestuff Reptr.* **34**, 42 (1945).
7. Landolt, A., *J. Soc. Dyers and Colourists* **64**, 93 (1948).
8. Lehmann, E., *Kolloid-Z.* **108**, 6–10 (1944).
9. Lindberg, J., TEXTILE RESEARCH JOURNAL **19**, 43 (1949).
10. Lindberg, J., and Gralén, N., TEXTILE RESEARCH JOURNAL **18**, 287 (1948).
11. Lindberg, J., Philips, B., and Gralén, N., *Nature* **162**, 458 (1948).
12. Makinson, K. R., TEXTILE RESEARCH JOURNAL **19**, 97 (1949).
13. Marshall, F. T., and Aulabaugh, S. T., TEXTILE RESEARCH JOURNAL **17**, 723 (1947).
14. McCleary, H. R., and Royer, G. L., TEXTILE RESEARCH JOURNAL **19**, 457 (1949) (this issue).
15. Mercer, E. H., *Nature* **155**, 573 (1945).

* Reg. U. S. Pat. Off.

16. Norman, D. P., Johnson, W. W. A., and Johnson, N. S., *Am. Dyestuff Reptr.* **37**, P838 (1948).
17. Speakman, J. B., Chamberlain, N. H., and Menkart, J., *J. Text. Inst.* **36**, T91 (1945).
18. Speakman, J. B., and Stott, E., *J. Text. Inst.* **22**, T339 (1931).
19. Speakman, J. B., Stott, E., and Chang, H., *J. Text. Inst.* **24**, T273 (1933).
20. Speakman, J. B., and Whewell, E., *J. Text. Inst.* **36**, T48 (1945).
21. Stock, C. R., and Salley, D. J., TEXTILE RESEARCH JOURNAL **19**, 41 (1949).
22. Whewell, C. S., and Wood, H. J., *J. Soc. Dyers and Colourists* **60**, 148 (1944).

(Manuscript received May 23, 1949.)

Shrinkage Control of Wool by Melamine Resins

Part II: Torsional Rigidity and Elastic Properties of Single Fibers

H. R. McCleary and G. L. Royer

American Cyanamid Company, Calco Chemical Division, Bound Brook, New Jersey

SINCE THE DEVELOPMENT of resin-treatment methods for making wool materials shrink-resistant, various opinions have been expressed by investigators in this field as to the mechanism by which resins act to control the shrinkage of wool. Two points of view have predominated—namely, (I) that the elastic properties of the fibers are changed by penetration of resin to the inside of individual fibers, and (II) that the surface frictional properties of the fibers are changed and that penetration of resin into single fibers, if it occurs at all, is only incidental. With respect to (I), it has been reported by Barr and Speakman [1] that a high degree of unshrinkability results from increasing the work of extension of wool fibers by treatments with cross-linking agents such as mercuric acetate or benzoquinone.

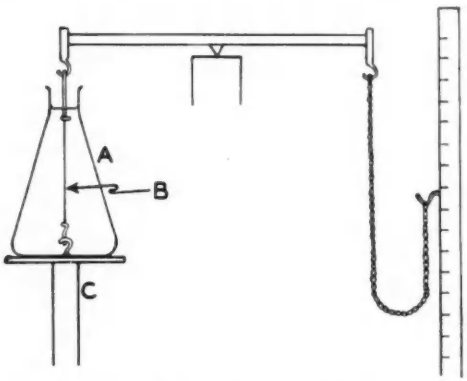

FIG. 1. *Fiber balance for obtaining stress-strain data on single fibers. A—flask filled with water. B—single fiber sealed to glass hooks. C—platform which may be raised or lowered.*

It is the purpose of this paper to present experimental evidence showing that treatment of wool fibers with methylated methylol melamine-type resin affects the surface portion of the fibers. Two types of data have been obtained on both treated and untreated single wool fibers—namely, stress-strain data and measurements of a function of torsional rigidity.

Experimental Procedures

Stress-Strain Measurements

The schematic diagram in Figure 1 shows the Chain-o-matic fiber balance which was used in extending single fibers. This balance is similar to the one described by Sookne and Rutherford [2]. The fiber was sealed to glass hooks with cellulose ace-

tate cement and hung from the left stirrup of the balance, the bottom hook being engaged by a hook in the bottom of the Erlenmeyer flask which was filled with water. The platform holding this flask was lowered manually to extend the fiber, while the chain loading mechanism, which was electronically controlled, adjusted itself to maintain balance and indicated the stress on the fiber.

The fibers used were South American wool extracted with both warm water and carbon tetrachloride. The fiber diameter was about 40 μ. The portions which were extended were 5- to 6-cm. sections cut midway between the roots and the tips of the fibers. After being sealed to glass hooks, all of the fibers were stored in water between extensions.

The procedure for extending the fibers was not one of constant rate of elongation, although extension was the independent variable. Instead, extension settings were made every 30 seconds to arbitrarily chosen points so that 14 to 16 points were obtained on the extension curve up to 30% extension and a like number of points were obtained on relaxation. Once a new fiber had been extended, subsequent extensions of this fiber were made at exactly the same extension values as in the first measurement. One complete cycle of extension and relaxation required 15 minutes. Data so obtained were plotted to give curves like those shown in Figure 2. In the experiments reported in this paper only the extension data were used. The area under each extension curve, measured by a planimeter, represents the work of extension and is expressed in units approximately equal to gram-centimeters. A new fiber was calibrated before treatment by making single extension and relaxation cycles on successive days until two successive work-of-extension measurements agreed within 2–3%. In general, the first extension was not counted, since it may remove the "kinks" from an initially unstretched fiber. Such calibrated fibers, sealed to glass hooks, were subjected to a given treatment and then extended at least twice to determine the effect of the treatment on work of extension.

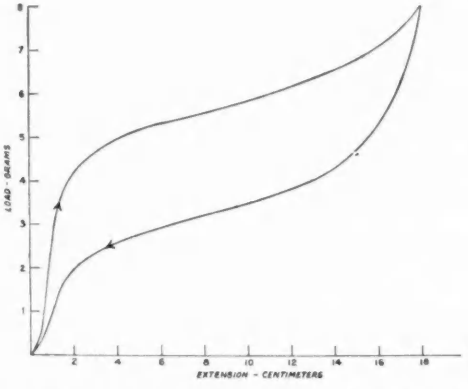

FIG. 2. *Extension-relaxation cycle for wool fiber of approximately 40 μ diameter and 6 cm. length, measured in water.*

Torsional Rigidity Measurements

In some earlier work by Speakman [3], results were reported on the torsional rigidity of single wool fibers at different relative humidities. For these determinations it was necessary to allow 24 hours for each fiber to reach equilibrium with an atmosphere of known humidity. In the experiments of the present paper it was desired to measure the rigidity of a fiber at a given, easily attainable moisture content, both before and after treatment. The apparatus used is unique in that it permits rapid successive measurements on individual fibers at

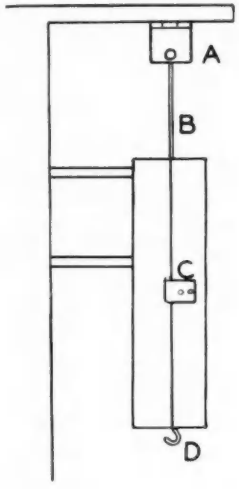

FIG. 3. *Fiber twisting apparatus—side view. A—hinged clamp supporting glass hook, B, to which fiber is attached. C—latch to hold "chimney" shut. D—lower glass hook to support torsion bar.*

100% relative humidity, with no time required to establish moisture equilibrium. The fibers used were identical with those used for stress-strain measurements and were attached to glass hooks, as described above. The fiber, which had been soaked in water for one day, was suspended in the center of a small "chimney" lined with felt soaked with water. The torsion bar hung below the chimney, while the fiber was completely surrounded by an atmosphere of 100% relative humidity. Figure 3 is a sketch of the chimney with the fiber suspended in place ready to receive the torsion bar on the bottom hook. Figure 4 is a top view, showing the hinged chimney opened to permit placing the fiber. Figure 5 is a picture of the apparatus. During a measurement, the entire set-up was covered by a small bell jar to exclude air currents.

The torsion bar, which must be non-magnetic, was made of aluminum. It was 4 cm. long and had a diameter of 2.25 mm. The bar, together with an aluminum center support, weighed 0.475 g.

The procedure for making measurements was as follows: The wool fiber on glass hooks, which had been standing in water so that it was thoroughly wet, was placed in the apparatus in the manner described above. A slotted piece of filter paper was placed on top of the chimney to eliminate vertical air currents. The aluminum bar was permitted to come to rest and then, by means of a manipulator, the bar was rotated through eight complete revolutions. It was then released and permitted to unwind through one-half cycle; the stop watch was started and the next complete period was measured, designated as Period I; the next one-half cycle was permitted to pass and then the next complete period was measured, designated as Period II. When the torsion bar had come to rest, the fiber was again twisted through eight revolutions of the bar in the direction opposite to that of the first twisting, and

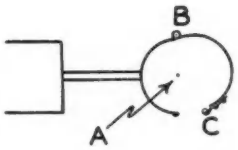

FIG. 4. *Fiber twisting apparatus, top view, showing "chimney" open. A—position of fiber. B—hinge. C—latch.*

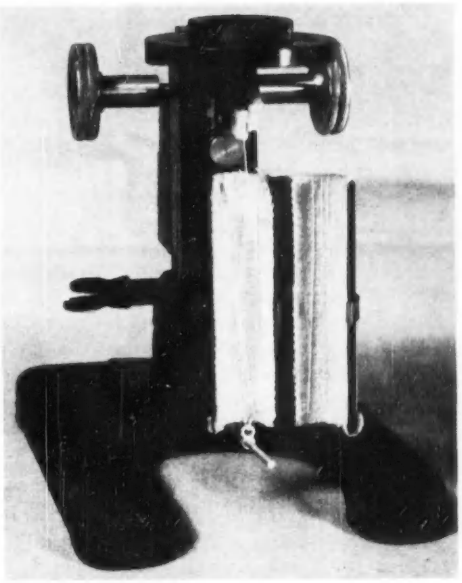

FIG. 5. *Fiber twisting apparatus showing torsion bar hung from fiber.*

Periods I and II were measured again. Results of successive measurements of this type are given in Table I for a typical fiber.

Single wool fibers in an atmosphere of 100% relative humidity show a very high degree of hysteresis on twisting. Thus, a particular fiber, which had been twisted by rotating the torsion bar through eight complete revolutions to the right, unwound to only 3.3 revolutions to the left of the rest point in the first half-cycle, followed by 2.7 revolutions to the right, 1.1 revolutions to the left, etc. In spite of this, however, the values for Periods I and II (Table I) are not very different.

In all of the twisting experiments reported in this paper, the value used for comparison of data is the

TABLE I. REPRODUCIBILITY OF TWIST-PERIOD MEASUREMENTS

Initial direction of twist →	Successive measurements of period (seconds)				
	Right	Left	Right	Left	Right
Period I	64.5	64.8	65.0	65.8	65.3
Period II	62.0	61.0	61.5	62.0	62.5

TABLE II. RESIN TREATMENT—EFFECT ON WORK OF EXTENSION

	Fiber No.	Work of extension (arbitrary units) Calibration	After treatment	% of calibration value
Resin-treated	63	89.5	85.4	95
Resin-treated	65	92.5	87.0	94
Control	60	86.4	82.3	95
Control	61	104.7	98.4	94

mean of at least three successive determinations, and Period I only has been used for such comparisons. No attempt has been made to calculate the rigidity of wool, η, as defined by Speakman [3]. In subsequent discussions of results, the term "twist period" is used to refer to the mean value for several measurements of Period I for a given fiber.

Results and Discussion

In order to determine the effect of LANASET * (a methylated methylol melamine-type) RESIN treatment on the work of extension of single wool fibers, the following experiments were carried out. Several calibrated fibers were resin-treated under conditions which were known to give good shrinkage control of wool materials. This involves treatment in a dilute aqueous solution of the resin, followed by drying at 80° to 90°C and curing at 145°–150°C. After treatment, the fibers were soaked in water for at least one day, after which extension measurements were made. Typical results are summarized in Table II, where data are included for control fibers which were heated only (not treated with resin). Each work-of-extension value is the average of at least two determinations.

The data in Table II show that this resin treatment had no effect on the work of extension of single wool fibers. It may be concluded, therefore, that shrinkage control of wool materials after such treatment is not due to changes in elastic properties of the individual fibers. Similar results have been reported recently by Stock and Salley [4].

Since no change in work of extension is brought about by resin treatment, it is probable that there is no resin in the interior of single fibers, but that it is in the epidermal layer. If it is present either as a tough film or as a tough packing at scale interstices, it might be expected that a treated fiber would be much more rigid (resistant to twisting) than an untreated fiber. This possibility was tested experimentally as follows:

Seven wool fibers mounted on glass hooks were calibrated for both work-of-extension and twist-period values. Previous experiments on wool fibers had shown that a single fiber may be calibrated according to both measurements since the determination of one has no effect on the calibration value of the other. Four of these fibers were treated in a LANASET RESIN bath. The remaining three fibers served as controls and were not treated in the resin bath. All fibers were then dried 10 minutes at 80°–85°C and cured 5 minutes at 145°–150°C. Table III gives the values for work of extension and twist period both before and after treatment. The measurements after treatment are listed in chronological order.

The results listed in Table III show, in agreement with data of Table II, that the work of extension is not changed by resin treatment. However,

* Reg. U. S. Pat. Off.

TABLE III. RESIN-TREATED FIBERS—WORK OF EXTENSION AND TWIST PERIODS

	Calibration values		Measurements after treatment (chronological order)				
			Twist period			Twist period	
Fiber No.	Work of extension	Twist period (sec.)	Sec.	% of Calibration	Work of extension	Sec.	% of Calibration
100	83.2	59.2	41.6	70%	82.7	47.5	80%
83	84.0	51.0	36.3	71%	84.6	44.7	88%
85	112.5	38.5	28.5	74%	112.0	34.1	89%
96	70.0	64.1	40.9	64%	72.0	50.5	79%
			(control fibers—heated and cured only)				
93	94.5	49.3	48.4	98%	90.0	48.8	99%
95	107.0	39.5	39.2	99%	107.0	39.6	100%
98	84.8	49.7	49.8	100%	82.3	50.7	102%

this treatment caused a marked increase in the rigidity (decrease of twist period) of single fibers. A further important point is that whereas the twist period immediately after treatment is only 65% to 75% of the calibration value, it increases to 80 to 90% of the calibration value after one extension of the fiber. These data indicate that resin treatment produces a tough shell of resin around the fiber, or seals the scales together, or causes a binding together of the scales in such a fashion as to cause a marked increase in rigidity of the fiber. If, however, such a fiber is stretched, the hard shell is fractured, or the seals between the scales are broken, and the fiber becomes less rigid. According to this picture, one would not expect resin treatment to cause any change in the work of extension because the first 1% or 2% of extension may fracture the resin coating and then, for the remainder of the extension, the fiber will behave like an untreated fiber.

According to Speakman [3], the rigidity, η, is related to the period, T, as follows:

$$\eta = k\left(\frac{1}{T^2}\right).$$

Therefore:

$$\frac{\eta_t}{\eta_u} = \frac{1}{(0.7)^2} = 2.0,$$

where the subscripts t and u refer to "treated" and "untreated" fibers, respectively, and the value 0.7 is the ratio of the twist periods for the treated versus the untreated fibers read from Table III. Hence, the rigidity of a resin-treated wool fiber (which has not been extended) is twice that of the untreated fiber.

Summary

It has been shown that treatment of single wool fibers with a methylated methylol melamine-type resin has no effect on the work of extension, but produces a marked increase in the torsional rigidity. Successive extensions of a treated fiber cause gradual decreases in the rigidity, which approaches that of the untreated fiber. These results are interpreted to mean that resin treatment affects only the surface portion of the wool fibers; this modified surface structure must, therefore, be chiefly responsible for the shrinkage resistance of wool fabrics.

The results of the present work are in complete agreement with those of Maresh and Royer [5], who made extensive microscopical studies of resin-treated wool fibers.

Acknowledgment

The authors acknowledge the help of their coworkers, especially that of Mrs. Elizabeth Watts and Mrs. Elizabeth Apisa, for obtaining much of the experimental data, and of Dr. R. H. Kienle for his support of this project.

Literature Cited

1. Barr, T., and Speakman, J. B., *J. Soc. Dyers and Colourists* **60**, 335 (1944).
2. Sookne, A. M., and Rutherford, H. A., *J. Research Natl. Bur. Standards* **31**, 25 (1943).
3. Speakman, J. B., *Trans. Faraday Soc.* **25**, 92 (1929).
4. Stock, C. R., and Salley, D. J., Textile Research Journal **19**, 41 (1949).
5. Maresh, C., and Royer, G. L., Textile Research Journal **19**, 449 (1949) (this issue).

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The Fungi Concerned in Fiber Deterioration

II: Their Ability to Decompose Cellulose*

P. B. Marsh,† K. Bollenbacher,† M. L. Butler,† and K. B. Raper‡

Abstract

On the basis of a survey of the literature and of test results here reported on more than 400 fungus isolates, cellulose-decomposing activity is shown to be very widely distributed but not universal among the fungi. It occurs with a high degree of regularity among isolates of certain genera—*e.g., Alternaria, Chaetomium, Curvularia, Fusarium, Gliomastix, Helminthosporium, Humicola, Memnoniella, Stachybotrys,* and *Trichoderma,* and probably with equal frequency in other genera which have not been so extensively studied. It occurs in a greater or smaller number of species in many of the several groups or subdivisions of the genus *Aspergillus* but apparently not in all of them, in a much smaller but rather widely diverse group of species in the genus *Penicillium,* apparently not at all in *Mucor, Rhizopus,* and other genera of the Mucoraceae, and probably only rarely among the actinomycetes.

Tests on *Cladosporium herbarum* show that the temperature optima for growth of isolates of this fungus are in general distinctly below the approximately 30°C used by several other workers in experiments with it; this fact is believed to account, at least in part, for the poor cellulose-decomposing activity shown by it in their tests. Tests on *Pullularia pullulans* have failed to detect cellulolytic activity.

Introduction

A knowledge of which fungi can decompose cellulose and which cannot is basic in the study of the microbiological deterioration of cellulosic fibers. The present paper is concerned with this subject. It is closely related in its experimental method to certain previous papers from the Department of Agriculture [9, 10, 17, 38] and to two recent contributions from the U. S. Army Quartermaster Corps [44, 46]. The principal technique used in the experiments reported in these papers and in the present work has involved the inoculation of cotton fabric with a pure culture of a fungus in the presence of mineral salts essential for fungus growth and the determination of change in strength of the fabric during a subsequent period of incubation. The basic method was described originally by Thom, Humfeld, and Holman [38] in 1934.

A not inconsiderable proportion of the information on cellulose decomposition by the fungi obtained before 1934 involved the use of somewhat unsatisfactory or even highly unsatisfactory experimental methods, with the consequence that many of the results obtained were inconclusive. White and coworkers [44], in the most important recent paper on the subject, dismissed this older information without discussion and, for the most part, without citation, apparently considering it to be too contradictory and unreliable to warrant use of the space required for its analysis.

Data from papers utilizing the general cotton fabric technique of Thom, Humfeld, and Holman [38] will be summarized in the paragraphs immediately following as an introduction to the data to be presented here. Earlier data and information obtained by other methods have been analyzed in the light of our own and other recent findings and have yielded several facts of significance not mentioned by other authors. These will be included in a Discussion section following the presentation of the experimental results, along with sufficient other pertinent information to provide a more complete historical background of the subject than previously has been available.

In 1934, Thom, Humfeld, and Holman [38] determined breaking-strength losses of cotton duck incubated with pure cultures of several fungi. Their technique consisted essentially of placing a steam-

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† Division of Cotton and Other Fiber Crops and Diseases, Bureau of Plant Industry, Soils, and Agricultural Engineering, U. S. Dept. of Agriculture, Beltsville, Maryland.

‡ Northern Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry, U. S. Dept. of Agriculture, Peoria, Illinois.

sterilized strip of cotton fabric on an agar medium in a culture vessel, inoculating the strip with a pure culture of a fungus, incubating the inoculated sample at a constant temperature for a period of several days, and finally determining its breaking strength on a pendulum-type Scott tester. Four *Chaetomium* isolates of known species and several of unnamed species, an isolate designated *Penicillium luteum*, and one of *Stachybotrys* were all definitely active.

In 1942, Greathouse, Klemme, and Barker [9] described a modification of the test method of Thom, Humfeld, and Holman [38] in which a piece of glass cloth and a water solution of mineral salts replaced the agar-mineral salts technique of the earlier work. They found losses in breaking strength of cotton fabric with "*Stachybotrys papyrogena*," *Chaetomium elatum*, *Hormodendrum* sp., *Alternaria* sp., and "*Metarrhizium* sp.," the last named producing the most rapid loss. Their "*Stachybotrys papyrogena*" was later found to be *Memnoniella echinata* [47]. Their *Hormodendrum*, as they were aware, is presumably synonymous with the *Cladosporium* of other workers. Their "*Metarrhizium* sp.," which was tentatively identified for them under this name by Dr. Charles Thom, was subsequently described as a new species by Pope [30] under the name "*Metarrhizium glutinosum*," but still later was shown by White and Downing [45] to be a misnamed isolate of *Myrothecium verrucaria*. Isolates of two species of the true genus *Metarrhizium* were tested later by White and coworkers [44] and found to be inactive in the degradation of cotton fabric.

Klemme, Greathouse, Bollenbacher, and Pope in 1945 [17] reported results of fabric culture bottle tests of 42 fungus isolates and noted the occurrence of active forms in the genera *Acremonium*, *Alternaria*, *Brachysporium*, *Cephalosporium*, *Fusarium* (12 isolates among 5 species), *Hormodendrum*, *Penicillium* (an isolate of the *P. luteum* series), *Stemphylium*, *Stysanus*, *Thielavia*, and *Trichoderma*. In the same year Greathouse and Ames [10] found each of the 16 *Chaetomium* species which they tested to be capable of bringing about strength loss of cotton duck in culture bottle tests with added mineral salts.

In a 1948 paper, White and coworkers [44] presented an account of pure-culture cotton fabric tests for cellulolytic activity of a considerably wider range of fungi than had been tested previously in any single laboratory. An earlier preliminary report which included parts of the same data, as well as other information, had appeared in December, 1945, under the authorship of Siu and White [34]. While these authors' work is too extensive to be summarized here and should be consulted in the original for details, some of their more important findings are condensed in Table I. To persons of limited experience with the fungi it might seem surprising and unnecessary for tests to be carried out on several isolates of a fungus to establish the behavior of the organism with respect to its ability to decompose cellulose. Those familiar with fungi, on the other hand, are well aware of the frequent existence of

TABLE I. SUMMARY OF SOME OF THE MORE IMPORTANT FINDINGS OF WHITE, DARBY, STECHERT, AND SANDERSON, 1948 [44]

Genus	Total number of isolates tested	Number of isolates found active
Acremonium	2	2
Alternaria	5	5
Aspergillus		
A. glaucus group	9	0
A. fumigatus group	7	4
A. nidulans group	7	0
A. ustus group	12	4
A. flavipes group	3	3
A. versicolor group	25	0
A. terreus group	7	7
A. niger group	29	1
A. wentii group	1	0
A. tamarii group	3	0
A. flavus-oryzae group	19	0
A. ochraceus group	2	0
Total, all groups	124	18
Botryodiplodia	3	1
Brachysporium	5	5
Chaetomium	15	15
Cladosporium	22	12
Curvularia	11	11
Fusarium	34	27
Gliocladium	8	4
Gliomastix	8	8
Humicola	7	7
Memnoniella	11	11
Metarrhizium	2	0
Mucoraceae	8	0
Myrothecium	8	7
Paecilomyces	9	0
Penicillium		
P. luteum group	11	5
P. citrinum group	20	4
All other groups	63	2
Total, all groups	94	11
Pestalotia	9	7
Phoma	6	6
Pullularia	5	0
Stachybotrys	4	4
Trichoderma	18	16
Verticillium	4	4

physiological variability in these organisms and will readily understand the necessity for this course of action.

White, Siu, and Reese [46] recently tested numerous isolates from the *Aspergillus niger* group for cellulolytic activity on cotton fabric. They concluded from their experiments that such activity is absent in the *A. niger* and *A. carbonarius* series of this group but is a fairly constant feature of the *A. luchuensis* series.

Materials and Methods

The test fabrics used were two 8-ounce cotton ducks designated in our laboratory as "M-2" and "Q" ducks. Breaking strength determinations for both original strength and strength after incubation with the fungus were made on 6 × 1-inch raveled strips on a pendulum-type Scott tester, the strips having been previously conditioned at approximately 70°F and 65% relative humidity for 24 or more hours before breaking. Early experiments were all done on the M-2 duck, the samples being prepared in small numbers from time to time and used directly as they came off the roll. An average break of 123 pounds was obtained for the original strength of this duck. Most of the work was carried out, however, on the Q duck. When work was started with the Q duck, 2,200 strips were cut at one time and were carefully randomized. The original fabric strength for this first randomized Q duck series was 105 pounds, a figure based on a total of 70 breaks which were made at a number of different times during the course of the work. When the first randomized series of Q duck was exhausted, another set was prepared, consisting of 1,800 strips. An original breaking strength of 111 pounds was obtained for this randomized set, this figure being an average from 75 breaks taken at intervals during the course of the work. The higher average strength for the second set is believed to have resulted from the fact that a leather facing was added on one surface of each of the jaws of the tester, resulting in better gripping of the samples. Application of the *t*-test led to the conclusion that strength differences required for significance at the 1% level between the control values of 105 and 111 pounds and the average values for the 5-break experimental sets were 7.7 and 8.8 pounds for the 2,200- and 1,800-strip randomized sets, respectively. Since small strength losses of nonbiological origin could conceivably occur from a variety of different causes, we have employed a conservative interpretation of the data and have claimed proof for cellulolytic activity only when the average strength loss was equal to or greater than 20%.

In order to obtain inoculum for the fabric tests, each culture was grown in a 250-cc. Erlenmeyer flask on a disc of filter paper resting on the surface of a 2% mineral salts agar or, in case insufficient or no growth was obtained on filter paper, on a medium containing the same mineral salts and agar plus 0.5% glucose and a water extract from raw cotton. The mineral salt composition of both these media, as well as of the medium used for the experimental fabric tests, was as follows:

NH_4NO_3	2.0 grams per liter
KH_2PO_4	1.8 grams per liter
K_2HPO_4	1.4 grams per liter
$MgSO_4 \cdot 7H_2O$	1.5 grams per liter

The cotton extract was prepared by kneading an immature raw cotton in water at room temperature. In the relatively few cases where cotton extract was used in the test bottles, specific mention of this fact is made in the table headings; in all such cases each liter of the final medium contained the extract from 100 g. of cotton.

The culture bottle tests recorded in Table II were carried out on 6 × 1-inch raveled strips which were wetted in water to which had been added 3 drops per liter of Igepal, a commercial, non-fungicidal wetting agent, and sterilized at 15 pounds steam pressure for 15 minutes. The strips were then planted with sterile precautions on the growth medium in 16-oz. French square bottles, inoculated with 1 cc. of an inoculum suspension, capped with a ¾-in. glass cloth air vent cap, incubated at approximately 30°C, harvested after 1-week and 2-week or only 2-week incubation periods, carefully washed in water to remove most of the superficial fungus material, dried at room temperature, conditioned, and broken.

Tests for which results are recorded in Tables III–VII were carried out according to the method used for the experiments of Table II except for the following deviations. Tests on *Cladosporium* (Table III) were carried out in constant temperature incubators at 15°, 20°, 25°, and 30°C. Special tests on several Aspergilli (Table IV) were carried out on the usual mineral salts agar but with 1% glucose added. Tests on fungi belonging to the order

Mucorales (Table V) were carried out on glass wicks of the kind described for this use by Greathouse, Klemme, and Barker [9], each fungus being tested in the presence of a mineral salt solution of the above-specified composition as well as with the same plus 1% glucose. The *Pullularia* isolates (Table VI) were tested by the method of Table II but with cotton extract and with cotton extract plus 1% glucose present in the mineral salts agar. The actinomycetes (Table VII) were tested on a salt medium the composition of which is specified in the table, "plus-glucose" and "minus-glucose" tests being run for each isolate.

In essentially all cases, and invariably in cases in which there seemed to be any question as to whether the fungus actually appearing on the test strips was the same as the organism in the original tube, microscopic mounts were made from the test specimens at the end of the incubation period. In cases in which no fungus growth appeared on the test strip, microscopic mounts were made to insure that lack of growth was not due to inhibition of the fungus by a bacterial contaminant. In the relatively few cases in which bacteria were found, the test results were discarded.

Experimental Results

The experiments to be described were carried out to a large extent on fungi found previously to be of common occurrence on fibers. This earlier information is contained in paper I of this series [23].

Table II presents results of tests for cellulose-decomposing activity carried out on a number of fungi on mineral salts agar at approximately 30°C. The various genera are arranged in alphabetical order in the table and will be discussed in that sequence. The various species of *Aspergillus* and *Penicillium* are arranged in the order which is followed in two recent manuals dealing with them [32, 39]. The experiments here reported on isolates of these genera from the Northern Regional Research Laboratory were planned with the thought in mind that these manuals might serve as a mycological foundation and a proof of identity for the cultures upon which physiological data are reported here. Many of the individual NRRL-numbered isolates tested have been described in these books [32, 39] and are included in a permanent fungus culture collection at Peoria. Certain of our experimental results are discussed below.

Alternaria. Ten of the 11 tested isolates of this fungus, which is of such common occurrence on fibers [23], were clearly active (Table II). The inactive isolate, *Alternaria* 6650, was still inactive when cultured at 15°, 20°, and 25°C. These results, supplementing previous observations by others [9, 17, 44], make it clear that isolates of *Alternaria* are, as a general rule, active in decomposing cellulose.

Aspergillus. Within this genus, White and coworkers [44] have found cellulolytic activity rather generally in four groups—namely, the *A. fumigatus*, *A. terreus*, *A. flavipes*, and *A. ustus* groups—with certain isolates being active also in a fifth group, the *A. niger* group. We have likewise found activity in each of these five groups, and have in addition noted one or more active isolates in the *A. clavatus*, *A. nidulans*, *A. ochraceus*, *A. tamarii*, and *A. versicolor* groups, thus having located activity in at least some isolates from ten out of the fourteen groups of the genus.

A. clavatus group. In this group 9 out of 14 isolates tested were definitely active. The active forms in general produced a smooth, even growth on the fabric, with the characteristic fruiting structure of the group.

A. glaucus group. None of the 3 isolates tested in this group was active.

A. fumigatus group. All of the 9 isolates tested in this group were found to be active. A uniform, heavy, greenish growth, characteristic of the fungus, was produced in all cases.

A. nidulans group. Active forms were found in 3 of the 5 species tested in this group. White and coworkers [44] had previously tested 7 isolates and found them all inactive.

A. ustus group. Four out of 5 isolates of *A. ustus* and both of the 2 isolates of *A. granulosus* tested were found to be active. Strength losses were in general more pronounced than those observed by White and coworkers [44].

A flavipes group. All of the 7 isolates of *A. flavipes* which were tested were clearly active, strength losses being greater than those noted for the 3 isolates tested by White and coworkers [44]. Growth was regularly uniform and heavy in appearance.

A. versicolor group. Of the 3 species tested, one was active—namely, *A. janus*—the 2 isolates of this active species showing good growth and fabric

TABLE II. CELLULOSE-DECOMPOSING ACTIVITY OF VARIOUS FUNGI AS INDICATED BY EXTENT OF TENDERING OF 8-OUNCE COTTON DUCK IN PURE CULTURE TESTS. TEST STRIPS INCUBATED ON MINERAL SALTS AGAR AT 30°C. EACH FIGURE IN TABLE REPRESENTS AN AVERAGE OBTAINED FROM FIVE REPLICATE STRIPS

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
Alternaria circinans ATCC 6650	ATCC	− 6	− 6
A. cucumerina ATCC 7794	ATCC	−38	−72
A. iridis ATCC 6657	ATCC	−35	−64
A. radicina ATCC 7891	ATCC	−39	−50
A. solani ATCC 6396	ATCC	−71	−86
A. solani ATCC 6662	ATCC	−67	−85
Alternaria sp. ATCC 9067	ATCC	−63	−70
Alternaria sp. BPI 101	Cotton locks, Raleigh, N. C.	−59	−85
Alternaria sp. BPI 52	Fabric, W. H. Weston, South Pacific exposure	−83	−98
Alternaria sp. BPI 71	Fabric, L. Heffner, above-ground Florida exposure	−65	−73
Alternaria sp. BPI 83.1	Fabric, C. Thom, awning, Long Island	−69	−90
Aspergillus clavatus Group			
A. clavatus NRRL–2	NRRL	−17	−25
A. clavatus NRRL–8	NRRL	−56	−70
A. clavatus NRRL–9	NRRL	−24	−38
A. clavatus ATCC 1007	ATCC	−14	−25
A. clavatus ATCC 9192 (Waksman #129)	ATCC	− 7	−13
A. clavatus ATCC 9198 (Waksman #164)	ATCC	−33	−63
A. clavatus ATCC 9598 (NCTC #978)	ATCC	−11	−26
A. clavatus ATCC 9599 (NCTC #5598A)	ATCC	0	0
A. clavatus ATCC 9600 (NCTC #3886)	ATCC	+ 1	− 9
A. clavatus ATCC 9601 (NCTC #5598)	ATCC	− 6	− 9
A. clavatus ATCC 9602 (NCTC #3887)	ATCC	−20	−27
A. clavatus ATCC 10058 (NRRL #4)	ATCC	−10	−17
A. giganteus NRRL–10	NRRL	−65	−71
A. giganteus ATCC 1031	ATCC	−51	−80
Aspergillus glaucus Group			
A. chevalieri ATCC 9908 (NRRL #77)	ATCC	− 7	− 6
A. amstelodami ATCC 1001 (Thom 4013)	ATCC	+ 7	+ 7
A. itaconicus ATCC 10021 (NRRL #161)	ATCC	− 1	− 5
Aspergillus fumigatus Group			
A. fumigatus BPI 37 (NRRL #A–1625)	Broom, Beltsville	−75	−100
A. fumigatus BPI 38 (NRRL #A–1626)	Soil	−72	−89
A. fumigatus BPI 39 (NRRL #A–1623)	Soil	−77	−86
A. fumigatus BPI 42 (NRRL #A–1624)	Soil-burial test strip	−62	−89
A. fumigatus BPI 65 (NRRL #A–1621)	Soil	−74	−100
A. fumigatus BPI 87 (NRRL #A–1620)	Laboratory contaminant, Beltsville	−62	−89
A. fumigatus ATCC 1028	ATCC	−40	−81
A. fumigatus ATCC 6285	ATCC	−70	−95
A. fumigatus ATCC 9197 (S. A. Waksman's strain 84)	ATCC	−33	−80
Aspergillus nidulans Group			
A. nidulans NRRL 193	NRRL	−30	−49
A. nidulans ATCC 10074 (NRRL #187)	ATCC	−23	−52
A. rugulosus NRRL 208	NRRL	−33	−53
A. variecolor NRRL 1858	NRRL	− 7	−10
A. variecolor ATCC 10067 (NRRL #212)	ATCC	−12	−28
A. caespitosus NRRL 1229	NRRL	− 1	− 1
A. caespitosus NRRL 1930	NRRL	− 1	− 5
A. unguis NRRL 217	NRRL	− 4	− 2
A. unguis ATCC 10073 (NRRL #216)	ATCC	+ 5	+ 2
Aspergillus ustus Group			
A. ustus BPI 10 (NRRL #A–1629)	Cardboard box, Beltsville	−61	−71

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
A. ustus BPI 11 (NRRL ✻A-1627)	Cardboard box, Beltsville	−54	−71
A. ustus BPI 48 (NRRL ✻A-1630)	Soil	−49	−72
A. ustus ATCC 10032 (From W. C. Tobie, Old Greenwich, Conn.)	ATCC	− 5	− 7
A. ustus var. *laevis* BPI 73 (NRRL ✻A-1619)	Fabric, O. R. Sutherland	−22	−62
A. granulosus NRRL 1931	NRRL	−32	−52
A. granulosus NRRL 1932	NRRL	−38	−57
Aspergillus flavipes Group			
A. flavipes BPI 36 (NRRL ✻A-1628)	Paper, Washington, D. C.	−49	−63
A. flavipes NRRL 289	NRRL	−53	−64
A. flavipes NRRL 297	NRRL	−51	−71
A. flavipes NRRL 528	NRRL	−59	−74
A. flavipes NRRL 1618	NRRL	−59	−67
A. flavipes NRRL 1723	NRRL	−50	−67
A. flavipes ATCC 1030 (NRRL ✻286)	ATCC	−34	−59
Aspergillus versicolor Group			
A. sydowi NRRL 242	NRRL	− 8	−11
A. sydowi NRRL 244	NRRL	+ 3	− 1
A. sydowi NRRL 246	NRRL	− 4	+ 2
A. sydowi NRRL 252	NRRL	− 3	− 2
A. sydowi NRRL 254	NRRL	−10	− 5
A. sydowi ATCC 9507	ATCC	−10	−11
A. janus NRRL 1936	NRRL	−60	−78
A. janus var. *brevis* NRRL 1935	NRRL	−25	−40
A. versicolor NRRL 225	NRRL	− 5	− 3
A. versicolor NRRL 226	NRRL	− 7	− 6
A. versicolor NRRL 230	NRRL	+ 2	− 3
A. versicolor NRRL 233	NRRL	− 7	0
A. versicolor ATCC 9577 (NRRL ✻238)	ATCC	− 5	−13
Aspergillus terreus Group			
A. terreus BPI 45 (NRRL ✻A-1622)	Fabric, L. Heffner, Florida exposure	−66	−97
A. terreus NRRL 267	NRRL	−56	−83
A. terreus NRRL 269	NRRL	−60	−77
A. terreus NRRL 274	NRRL	−63	−81
A. terreus ATCC 10020 (NRRL ✻1960)	ATCC	−51	−79
A. terreus ATCC 10029 (NRRL ✻265)	ATCC	−66	−73
A. terreus ATCC 10071 (NRRL ✻255)	ATCC	−66	−83
A. carneus NRRL 1927	NRRL	−53	−75
A. carneus NRRL 1928	NRRL	−49	−68
A. niveus NRRL 1955	NRRL	−49	−70
Aspergillus candidus Group			
A. candidus NRRL 304	NRRL	+12	+ 1
A. candidus NRRL 306	NRRL	+ 8	+ 7
A. candidus NRRL 307	NRRL	+13	− 7
A. candidus NRRL 308	NRRL	+ 8	− 1
A. candidus NRRL 312	NRRL	+14	− 5
A. candidus NRRL 313	NRRL	+13	+ 1
Aspergillus niger Group			
A. niger ATCC 1015 (Thom's TC 167)	ATCC	− 5	− 7
A. niger ATCC 6277 (Thom's 215-5373.16)	ATCC	− 2	− 5
A. niger TC 215-4247 (Thom & Church Collection)	BPI	− 4	− 5
A. foetidus NRRL 341	NRRL	− 5	− 3
A. phoenicis NRRL 1956	NRRL	+ 1	− 1
A. niger mut. *cinnamomeus* NRRL 348	NRRL	− 4	+ 2
A. niger mut. *cinnamomeus* ATCC 1027 (Thom's 204-35346)	ATCC	+ 8	+ 5

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
A. carbonarius ATCC 1025 (NRRL ✱ 369)	ATCC	− 7	− 3
A. luchuensis ATCC 1035 (Thom's ✱218–4828.8)	ATCC	− 5	− 5
A. luchuensis NRRL 356	NRRL	− 9	− 8
A. japonicus NRRL 358	NRRL	−21	−30
A. japonicus NRRL 359	NRRL	−30	−45
A. violacea-fuscus NRRL 360	NRRL	− 1	−12
Aspergillus wentii Group			
A. alliaceus ATCC 1024 (Thom's strain 200–5506)	ATCC	0	−12
A. alliaceus ATCC 10060 (NRRL ✱315)	ATCC	− 7	−10
A. avenaceus NRRL 517	NRRL	− 9	−15
A. wentii NRRL 376	NRRL	− 2	− 3
A. wentii NRRL 377	NRRL	− 2	− 4
A. wentii NRRL 378	NRRL	− 3	− 3
A. wentii NRRL 380	NRRL	− 4	− 2
A. wentii NRRL 382	NRRL	− 4	+ 2
Aspergillus tamarii Group			
A. terricola ATCC 10062 (NRRL ✱424)	ATCC	−12	−44
A. tamarii NRRL 428	NRRL	− 2	−11
A. tamarii NRRL 429	NRRL	− 8	−17
A. tamarii NRRL 437	NRRL	− 7	−16
A. tamarii NRRL 438	NRRL	− 9	− 9
A. tamarii NRRL 439	NRRL	0	− 4
A. tamarii NRRL 443	NRRL	− 2	− 5
A. tamarii ATCC 1005 (NRRL ✱427)	ATCC	−12	− 7
Aspergillus flavus—oryzae Group			
A. oryzae NRRL 448	NRRL	− 8	− 7
A. oryzae ATCC 10063 (NRRL ✱458)	ATCC	− 9	− 5
A. flavus NRRL 483	NRRL	− 6	−13
A. flavus NRRL 486	NRRL	− 9	−11
A. flavus NRRL 487	NRRL	− 3	− 6
A. flavus ATCC 7252	ATCC	− 2	− 3
A. flavus ATCC 9296 (from Waksman ✱137)	ATCC	−17	−18
A. flavus ATCC 9807 (M. F. Day's strain ✱42)	ATCC	− 6	− 7
A. flavus ATCC 10124 (NRRL ✱484)	ATCC	− 7	− 4
A. parasiticus ATCC 1018 (NRRL ✱502)	ATCC	− 6	− 7
A. effusus ATCC 1010 (NRRL ✱506)	ATCC	− 4	0
Aspergillus ochraceus Group			
A. sulphureus ATCC 1032 (NRRL ✱386)	ATCC	−33	−69
A. sulphureus ATCC 9538 (NRRL ✱286)	ATCC	− 6	− 8
A. quercinus NRRL 387	NRRL	−18	−35
A. quercinus NRRL 391	NRRL	−24	−52
A. quercinus NRRL 393	NRRL	−29	−53
A. sclerotiorum NRRL 415	NRRL	− 8	− 6
A. ochraceus NRRL 410	NRRL	−33	−49
A. ochraceus NRRL 413	NRRL	−17	−42
A. ochraceus NRRL 417	NRRL	−30	−52
A. ochraceus ATCC 1009 (Thom 260–4399)	ATCC	− 7	−25
Anthostomella sp. ATCC 7888	ATCC	−87	−100
Ashbya gossypii ATCC 8717	ATCC	0	+ 1
Byssochlamys fulva NRRL 1125	NRRL	—	− 2
Cephalothecium roseum ATCC 8685	ATCC	−15	−52
Circinella spinosa ATCC 9025	ATCC	− 1	− 3
Circinella sydowi ATCC 9023	ATCC	− 1	− 2
Coccosporium sp.	W. W. Diehl	−30	−85
Corticium vagum ATCC 6221	ATCC	−13	−55
Cunninghamella verticillata ATCC ✱8985	ATCC	+ 3	+ 3

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
Cunninghamella verticillata ATCC #8983	ATCC	+ 4	+ 1
Curvularia sp. BPI 75	Fabric, C. Thom, awning, Long Island	−84	−88
Curvularia sp. 1592	H. S. Sherwin	−80	−89
Curvularia sp. 2057	H. S. Sherwin	−71	−92
Curvularia sp. 3048	H. S. Sherwin	−62	−83
Curvularia sp. 3202	H. S. Sherwin	−75	−91
Diplodia sp. BPI 120	Cotton boll, Florence, S. C.	−13	−22
Diplodia sp. BPI 121	Cotton lock, Florence, S. C.	−30	−63
Diplodia natalensis ATCC 9055	ATCC	−46	−74
Endoconidiophora sp. 83	L. P. McCulloch	+ 2	− 1
Fusarium avenaceum ATCC 8150	ATCC	−61	−78
F. conglutinans var. *majus* 208	W. D. McClellan	−65	−84
F. lateritium	W. C. Snyder, apple	−42	−68
F. moniliforme BPI 102	Cotton locks, Raleigh, N. C.	−54	−100
F. moniliforme BPI 104	Cotton locks, Rocky Mount, N. C.	−61	−83
F. nivale	W. C. Snyder, barley	−23	−47
F. oxysporum 177	W. D. McClellan	−61	−73
F. roseum f. *cerealis*	W. C. Snyder, wheat	−54	−74
F. solani f. *cucurbitae*	W. C. Snyder, squash	+ 6	+ 3
F. sulphureum ATCC 7642	ATCC	+ 4	− 1
F. tricinctum	W. C. Snyder, grass	−54	−71
F. vasinfectum ATCC 7808	ATCC	−51	−67
F. zonatum ATCC 7624	ATCC	−57	−68
Fusarium sp.	A. L. Smith, yellowed cotton	−55	−64
Fusarium sp. BPI 72	Fabric, L. Heffner, Florida exposure	−50	−71
Fusarium sp. BPI 79	Soil-burial test strip	−61	−77
Gliocladium catenulatum NRRL 1091	NRRL	—	−72
G. deliquescens NRRL 1086	NRRL	—	−82
G. roseum NRRL 1085	NRRL	—	−67
G. vermoeseni NRRL 1752	NRRL	—	−43
Gliocladium sp. 4	Chemical Warfare Service	−65	−85
Glomerella sp. BPI 105	Cotton locks, Raleigh, N. C.	−74	−100
Glomerella fusiforme ATCC 9552	ATCC	−80	−85
Guignardia bidwellii ATCC 9559	ATCC	− 4	− 5
Guignardia bidwellii ATCC 9560	ATCC	− 5	− 5
Helminthosporium avenae 46–29–1c	A. L. Robert, oats	−47	−74
H. carposaprum #8	L. P. McCulloch	−42	−63
H. carposaprum	F. Pollack, tomato	−54	−71
H. gramineum 46–28–1c	A. L. Robert, barley	−45	−85
H. halodes	F. Pollack, tomato	−71	−82
H. tritici-vulgaris 45–2–1a	A. L. Robert, wheat	−71	−79
H. tritici-repentis 45–4–1c	A. L. Robert, quack-grass	−38	−63
H. turcicum 46–C–1	A. L. Robert, corn	−50	−83
Helminthosporium sp. BPI 76	Fabric, L. Heffner, Florida exposure	−58	−91
Hendersonia sp. BPI 93	Fabric, L. Heffner, Florida exposure	−72	−94
Humicola sp. BPI 1	Laboratory contaminant	−100	−100
Humicola sp. BPI 5	Fabric, C. P. Hartley	−95	−100
Humicola sp. BPI 8	Fabric, W. H. Weston, S. Pacific exposure	−87	−100
Humicola sp. BPI 9	Fabric, W. H. Weston, S. Pacific exposure	−94	−100
Humicola sp. BPI 19	Soil-burial test strip	−85	−100
Humicola sp. BPI 47	Fabric, A. F. Verrall, Mississippi exposure	−94	−100
Humicola sp. BPI 54	Fabric, W. H. Weston, S. Pacific exposure	−80	—

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
Humicola sp. BPI 55	Fabric, W. H. Weston, S. Pacific exposure	−94	−100
Humicola sp. 63	S. A. Waksman	−77	−100
Monascus ruber #7	W. W. Diehl (Hodges)	−4	−5
Monascus purpureus 6405	ATCC	−1	+1
Monotospora daleae ATCC 6725	ATCC	−100	−100
Neurospora crassa ATCC 9278	ATCC	−73	−100
Neurospora crassa ATCC 9683	ATCC	−84	−100
Neurospora crassa ATCC 9277	ATCC	−55	−82
Neurospora tetrasperma ATCC 6484	ATCC	−93	−100
Paecilomyces varioti NRRL 115	NRRL	—	−1
Penicillium javanicum Series			
P. javanicum van Beyma NRRL 707	NRRL	—	−84
P. brefeldianum Dodge NRRL 2083	NRRL	—	−90
P. levitum Klebahn NRRL 705	NRRL	—	−93
P. thomii Series			
P. thomii Maire NRRL 1640	NRRL	—	−24
P. sclerotiorum van Beyma NRRL 2074	NRRL	—	−3
P. lapidosum Raper and Fennell NRRL 718	NRRL	—	−22
P. turbatum Westling NRRL 757	NRRL	—	−4
P. frequentans Series			
P. frequentans Westling NRRL 1915	NRRL	—	−3
P. spinulosum Thom NRRL 1750	NRRL	−4	−4
P. spinulosum ATCC 9123	ATCC	−4	−5
P. lividum Series			
P. lividum Westling NRRL 754	NRRL	—	+1
P. aurantio-violaceum Biourge NRRL 760	NRRL	—	−2
P. trzebinskii Zaleski NRRL 731	NRRL	—	−2
P. implicatum Series			
P. multicolor Grigorieva-Manеilova and Poradie-lova NRRL 2058	NRRL	—	−90
P. implicatum Biourge NRRL 2054	NRRL	—	+2
P. decumbens Series			
P. chermesinum Biourge NRRL 2049	NRRL	—	+6
P. decumbens Thom NRRL 742	NRRL	—	−2
P. fellutanum Biourge NRRL 746	NRRL	—	+4
P. citreo-viride Biourge NRRL 2046	NRRL	—	0
P. restrictum Series			
P. restrictum Gilman and Abbott NRRL 1748	NRRL	—	+4
P. adametzi Series			
P. adametzi Zaleski NRRL 737	NRRL	—	−7
P. terlikowskii Zaleski NRRL 2067	NRRL	—	−12
Ramigena Series			
P. capsulatum Raper and Fennell NRRL 2056	NRRL	—	−5
P. cyaneum (B. and S.) Biourge NRRL 775	NRRL	—	+6
P. waksmani Zaleski NRRL 777	NRRL	—	−21
Carpenteles Series			
P. asperum (Shear) Raper and Thom NRRL 715	NRRL	—	+1
P. baarnense van Beyma NRRL 2086	NRRL	—	+2
P. egyptiacum van Beyma NRRL 2090	NRRL	—	−17
P. raistrickii Series			
P. raistrickii Smith NRRL 2039	NRRL	—	+4

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
P. pulvillorum Turfitt NRRL 2082	NRRL	—	−89
P. soppi Zaleski NRRL 2023	NRRL	—	−12
P. lilacinum Series			
P. lilacinum Thom NRRL 895	NRRL	—	+ 1
Spicaria violacea Abbott NRRL 901	NRRL	—	− 6
P. humuli van Beyma NRRL 872	NRRL	—	+ 4
P. janthinellum Series			
P. janthinellum Biourge NRRL 2016	NRRL	—	−93
P. simplicissimum (Oud.) Thom NRRL 902	NRRL	—	−81
P. simplicissimum BPI 15 (NRRL A-1613)	Fabric, H. Rutherford, New Orleans exposure	−71	−94
P. simplicissimum BPI 33 (NRRL A-1618)	Soil-burial test strip	−50	−100
P. simplicissimum BPI 49 (NRRL A-1617)	Soil	−73	−94
P. simplicissimum BPI 68 (NRRL A-1614)	Laboratory contaminant	−80	−100
P. simplicissimum BPI 84 (NRRL A-1612)	Soil-burial test strip	−80	−96
P. ochro-chloron Biourge NRRL 926	NRRL	—	−93
P. miczynskii Zaleski NRRL 1077	NRRL	—	−16
P. canescens Series			
P. canescens Sopp NRRL 910	NRRL	—	−21
P. jenseni Zaleski NRRL 909	NRRL	—	− 1
P. nigricans Series			
P. nigricans (Bainier) Thom NRRL 915	NRRL	—	− 3
P. nigricans ATCC 10115	ATCC	− 7	−17
P. melinii Thom NRRL 2041	NRRL	—	0
P. raciborskii Zaleski NRRL 2150	NRRL	—	− 5
P. citrinum Series			
P. corylophilum Dierckx NRRL 802	NRRL	—	+ 4
P. citrinum Thom NRRL 806	NRRL	—	−35
P. citrinum ATCC 8506 (NRRL 1843)	ATCC	− 2	− 5
P. steckii Zaleski NRRL 2140	NRRL	—	−21
P. chrysogenum Series			
P. chrysogenum Thom NRRL 807	NRRL	—	−14
P. chrysogenum NRRL 1951	NRRL	—	−14
P. chrysogenum ATCC 6203	ATCC	−12	−26
P. notatum Westling NRRL 821	NRRL	—	−19
P. oxalicum Series			
P. oxalicum Currie and Thom NRRL 790	NRRL	—	−94
P. atramentosum Thom NRRL 795	NRRL	—	−14
P. digitatum Series			
P. digitatum Saccardo NRRL 786	NRRL	—	−10
P. digitatum ATCC 10030	ATCC	+ 3	+ 4
P. roqueforti Series			
P. roqueforti Thom NRRL 849	NRRL	—	+ 3
P. roqueforti ATCC 6989	ATCC	0	− 1
P. casei Staub NRRL 844	NRRL	—	+ 1
P. brevi-compactum Series			
P. brevi-compactum Dierckx NRRL 2011	NRRL	—	− 6
P. brevi-compactum ATCC 9056 (NRRL 864)	ATCC	+ 2	− 1
P. stoloniferum Thom NRRL 859	NRRL	—	−29
P. paxilli Bainier NRRL 2008	NRRL	—	− 7
P. camemberti Series			
P. caseicolum Bainier NRRL 875	NRRL	—	−14

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
P. camemberti Thom NRRL 877	NRRL	—	− 15
P. camemberti ATCC 6985	ATCC	+ 5	+ 6
P. commune Series			
P. lanosum Westling NRRL 2009	NRRL	—	− 3
P. lanoso-viride Thom NRRL 879	NRRL	—	0
P. lanoso-coeruleum Thom NRRL 888	NRRL	—	− 22
P. biforme Thom NRRL 885	NRRL	—	+ 1
P. commune Thom NRRL 890	NRRL	—	− 2
P. terrestre Series			
P. terrestre Jensen NRRL 934	NRRL	—	− 5
P. terrestre ATCC 10122	ATCC	+ 1	+ 5
P. solitum Westling NRRL 937	NRRL	—	− 4
P. resticulosum Birkinshaw, Raistrick, and Smith NRRL 2021	NRRL	—	− 3
P. pallidum Series			
P. pallidum Smith NRRL 2037	NRRL	—	+ 1
P. putterillii Thom NRRL 2024	NRRL	—	+ 4
P. lavendulum Raper and Fennell NRRL 2146	NRRL	—	− 1
P. gladioli Series			
P. gladioli Machacek NRRL 939	NRRL	—	− 4
P. ochraceum Series			
P. ochraceum (Bainier) Thom NRRL 871	NRRL	—	− 4
P. carneo-luteseens Smith NRRL 2035	NRRL	—	0
P. viridicatum Series			
P. viridicatum Westling NRRL 963	NRRL	—	+ 4
P. viridicatum ATCC 10117	ATCC	− 3	− 5
P. olivino-viride Biourge NRRL 2028	NRRL	—	+ 1
P. palitans Westling NRRL 2033	NRRL	—	0
P. cyclopium Series			
P. cyclopium Westling NRRL 1899	NRRL	—	+ 5
P. cyclopium Westling var. *echinulatum* Raper and Thom NRRL 1151	NRRL	—	− 7
P. cyclopium ATCC 8507	ATCC	+ 3	+ 3
P. puberulum ATCC 8508	ATCC	+ 4	+ 4
P. martensii Biourge NRRL 2027	NRRL	—	0
P. expansum Series			
P. expansum Link NRRL 976	NRRL	—	+ 4
P. expansum ATCC 1117	ATCC	− 5	− 18
P. crustosum Thom NRRL 968	NRRL	—	+ 2
P. italicum Series			
P. italicum Wehmer NRRL 983	NRRL	—	− 8
P. italicum ATCC 1122	ATCC	− 8	− 5
P. urticae Series			
P. urticae Bainier NRRL 989	NRRL	—	− 26
P. granulatum Series			
P. corymbiferum Westling NRRL 2032	NRRL	—	− 9
P. claviforme Series			
P. claviforme Bainier NRRL 2031	NRRL	—	− 16
P. clavigerum Demelius NRRL 1003	NRRL	—	− 2
P. luteum Series			
P. stipitatum Thom NRRL 1006	NRRL	—	− 77

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
P. vermiculatum Dangeard NRRL 1019	NRRL	—	−77
P. wortmanni Klocker NRRL 1017	NRRL	—	− 6
P. helicum Raper and Fennell NRRL 2106	NRRL	—	−54
P. rotundum Raper and Fennell NRRL 2107	NRRL	—	− 4
P. bacillosporum Swift NRRL 1025	NRRL	—	−16
P. avellaneum Thom and Turesson NRRL 1938	NRRL	—	− 1
P. avellaneum ATCC 1019 (NRRL 1005)	ATCC	− 3	− 1
P. luteum series (nonascosporic) BPI 66 (NRRL A-1616)	Laboratory contaminant	−64	−88
P. luteum series (nonascosporic) BPI 85.1 (NRRL A-1615)	Fabric, Beltsville	−56	−83
P. luteum series (nonascosporic) ATCC 1124 (C. Thom)	ATCC	−50	−84
P. luteum series (nonascosporic) ATCC 9644 (W. H. Weston)	ATCC	−55	−85
P. luteum series (nonascosporic) ATCC 9776 (P. B. Marsh)	ATCC	+ 1	−15
P. luteum series (nonascosporic) ATCC 10125 (NRRL 1015)	ATCC	−81	−100
P. luteum Zukal NRRL 1010	NRRL	—	− 1
P. striatum Raper and Fennell NRRL 717	NRRL	—	− 4
P. duclauxii Series			
P. duclauxii Delacroix NRRL 1030	NRRL	—	− 4
P. funiculosum Series			
P. funiculosum Thom NRRL 1032a	NRRL	—	−91
P. funiculosum Thom NRRL 1768	NRRL	—	−89
P. funiculosum ATCC 9846 (NRRL 1035)	ATCC	+ 2	− 4
P. verruculosum Peyronel NRRL 1050	NRRL	—	−61
P. islandicum Sopp NRRL 1036	NRRL	—	−10
P. varians Smith NRRL 2096	NRRL	—	−18
P. piceum Raper and Fennell NRRL 1051	NRRL	—	− 2
P. purpurogenum Series			
P. purpurogenum Stoll var. *rubri-sclerotium* Thom NRRL 1066	NRRL	—	−96
P. purpurogenum ATCC 9777 (NRRL 1061)	ATCC	+ 1	−23
P. rubrum Stoll NRRL 1062	NRRL	—	0
P. aculeatum Raper and Fennell NRRL 2129	NRRL	—	−85
P. variable Sopp NRRL 1048	NRRL	—	− 4
P. sanguineum ATCC 10064 (NRRL 1059)	ATCC	− 4	− 8
P. rugulosum Series			
P. rugulosum ATCC 10128 (NRRL 1045)	ATCC	0	− 8
P. tardum Thom NRRL 1073	NRRL	—	0
P. diversum Raper and Fennell NRRL 2121	NRRL	—	+ 2
P. diversum var. *aurcum* Raper and Fennell NRRL 1074	NRRL	—	0
P. herquei Series			
P. herquei Bainier NRRL 1040	NRRL	—	−12
P. novae-zealandiae van Beyma NRRL 2128	NRRL	—	−14
Phialophora malorum 92	L. P. McCulloch	−67	−83
Phoma glomerata ATCC 6735	ATCC	−60	−83
Phoma betae ATCC 6504	ATCC	+ 1	0
Phoma destructiva ATCC 9641	ATCC	−67	−86
Pullularia pullulans J364	W. L. White	+ 5	+ 3
Pullularia pullulans PQMD 72h	W. L. White	+ 2	0
Pullularia pullulans Weber F 44	W. L. White	+ 3	− 1
Sclerotinia fructicola 28	L. P. McCulloch	−11	−28

TABLE II (*Continued*)

Fungus name and isolate number	Source	Percent change in strength 1 wk.	2 wk.
Scopulariopsis brevicaulis NRRL 1096	NRRL	—	−44
Sordaria fimicola ATCC 9985	ATCC	−100	−100
Sporotrichum citri 85	L. P. McCulloch	−30	−89
Stachybotrys lobulata ATCC 7436	ATCC	−60	−66
Stachybotrys atra BPI 7	Fabric, C. Hartley	−85	−100
Stachybotrys atra BPI 35	Air contaminant, Beltsville	−63	−79
Syncephalastrum fuliginosum ATCC 8981	ATCC	0	−5
Thamnidium elegans ATCC 8997	ATCC	+8	+5
Thielavia sp. BPI 18	Fabric, H. Rutherford, New Orleans exposure	−85	−100
Thielavia sp. BPI 46	Fabric, A. F. Verrall, Mississippi exposure	−92	−100
Torula convoluta PQMD 95	Sewing of shoe, Phila. QMD	−89	−100
Tritirachium roseum	W. W. Diehl	−1	−3
Trichoderma lignorum ATCC 8678	ATCC	−67	−83
T. viride ATCC 10046	ATCC	−56	−76
Trichoderma sp. 12	Chemical Warfare Service	−75	−94
Trichoderma sp. BPI 29	Soil-burial test strip	−71	−91
Trichoderma sp. BPI 43	Soil-burial test strip	−72	−98
Trichoderma sp. 53	Chemical Warfare Service	−76	−94
Trichoderma sp. BPI 56	Fabric, Phila. Navy Yard	−55	−93
Verticillium sp. ATCC 7611	ATCC	−68	−71
Zygorhynchus moelleri ATCC 1208	ATCC	0	−1

TABLE III. EFFECT OF TEMPERATURE ON ACTIVITY OF ISOLATES OF *Cladosporium* SP. IN TENDERING 8-OUNCE COTTON DUCK IN PURE-CULTURE TESTS. TEST STRIPS INCUBATED ON MINERAL SALTS AGAR. ALL CULTURES EXCEPT ATCC 6506 AND ✱48 ISOLATED FROM FIELD-WEATHERED COTTON. ISOLATE ✱48 RECEIVED FROM L. P. MCCULLOCH

Fungi	Incubation time	Percent change in strength at 15°C	20°C	25°C	30°C
Cladosporium herbarum ATCC 6506	1 week	−29	−20	−5	+1
Cladosporium herbarum ATCC 6506	2 weeks	−37	−31	−7	+6
Cladosporium herbarum ✱48	1 week	−12	−31	−7	−5
Cladosporium herbarum ✱48	2 weeks	−17	−51	−15	+5
Cladosporium herbarum BPI 106	1 week	−25	−48	−57	−22
Cladosporium herbarum BPI 106	2 weeks	−53	−80	−87	−24
Cladosporium herbarum BPI 107	1 week	−8	−21	−14	−5
Cladosporium herbarum BPI 107	2 weeks	−50	−46	−43	−11
Cladosporium herbarum BPI 108	1 week	−11	−44	−44	−3
Cladosporium herbarum BPI 108	2 weeks	−51	−68	−76	+4
Cladosporium herbarum BPI 109	1 week	−22	−40	−40	−8
Cladosporium herbarum BPI 109	2 weeks	−68	−77	−68	−5
Cladosporium herbarum BPI 110	1 week	−25	−53	−71	−20
Cladosporium herbarum BPI 110	2 weeks	−54	−78	−87	−15
Cladosporium herbarum BPI 111	1 week	−21	−31	−22	−20
Cladosporium herbarum BPI 111	2 weeks	−31	−53	−36	−17
Cladosporium herbarum BPI 112	1 week	−26	−54	−65	−20
Cladosporium herbarum BPI 112	2 weeks	−62	−83	−84	−35
Cladosporium herbarum BPI 113	1 week	−36	−60	−74	−60
Cladosporium herbarum BPI 113	2 weeks	−71	−83	−85	−89

TABLE IV. RESULTS OF EXPERIMENT TO CHECK ADEQUACY OF BASAL SALTS MEDIUM FOR GROWTH OF SEVERAL ASPERGILLI PREVIOUSLY FOUND INACTIVE (TABLE II) AND POSSIBILITY OF FABRIC TENDERING BY THESE FUNGI IN THE PRESENCE OF GLUCOSE. TWO WEEKS' EXPOSURE, 30°C, GLASS WICK TECHNIQUE WITH 1% GLUCOSE ADDED TO BASAL SALTS MEDIUM. ALL ISOLATES EXCEPT THOSE MARKED WITH ASTERISK (*) WERE INACTIVE ON MINERAL SALTS AGAR

Fungi	Growth rating	Percent change in strength
**Aspergillus clavatus* NRRL 8	V. Heavy	−11
**Aspergillus nidulans* NRRL 193	V. Heavy	−31
**Aspergillus flavipes* NRRL 528	V. Heavy	−80
Aspergillus versicolor NRRL 230	Heavy	+ 8
Aspergillus versicolor NRRL 244	Heavy	+ 10
**Aspergillus terreus* ATCC 10020	V. Heavy	−76
Aspergillus candidus NRRL 308	Heavy	+ 4
Aspergillus candidus NRRL 313	Heavy	+ 3
Aspergillus alliaceus ATCC 1024	Heavy	+ 9
Aspergillus avenaceus NRRL 517	Heavy	+ 2
Aspergillus wentii NRRL 382	V. Heavy	+ 1
Aspergillus tamarii NRRL 428	V. Heavy	− 3
Aspergillus tamarii NRRL 429	V. Heavy	− 3
Aspergillus tamarii NRRL 438	V. Heavy	+ 1
Aspergillus oryzae NRRL 448	V. Heavy	+ 7
Aspergillus flavus NRRL 483	V. Heavy	+ 5
Aspergillus flavus NRRL 486	V. Heavy	+ 6

TABLE V. ACTIVITY OF SEVERAL ISOLATES OF SPECIES FROM THE MUCORACEAE IN TENDERING 8-OUNCE COTTON DUCK IN PURE CULTURE TESTS WITH AND WITHOUT GLUCOSE ADDED TO BASAL MINERAL SALTS MEDIUM. TWO WEEKS' EXPOSURE, 30°C, GLASS WICK TECHNIQUE.* ALL ISOLATES WERE OBTAINED FROM DR. V. M. CUTTER

Fungi	Percent change in strength: Mineral salts only	Mineral salts–1% glucose
Absidia sp. 250	+11	+ 2
Absidia sp. 861	+ 2	− 2
Circinella sydowi 595	+ 3	− 5
Circinella simplex 844	− 1	+ 3
Cunninghamella elegans 555	+ 5	0
Cunninghamella echinulata 743	+ 6	+ 8
Mortierella Bainieri 819	+ 7	+ 7
Micromucor sp. 826	+ 5	+ 3
Mucor varians 596	− 7	− 3
Mucor hiemalis 732	+ 5	+ 4
Mucor abundans 620	− 3	− 1
Rhizopus arrhizus 493	+ 3	− 4
Rhizopus nigricans 806	0	− 2
Rhizopus microsporus 437	+ 9	− 3
Syncephalastrum racemosum 554	+ 3	− 2
Syncephalastrum racemosum 104	− 1	− 8
Tieghemella glauca 156	+ 6	+10
Zygorhynchus sp. 840	+ 3	+ 4

* In all cases moderate to heavy growth occurred on the glucose-containing medium.

TABLE VI. ACTIVITY OF SEVERAL ISOLATES OF *Pullularia* IN TENDERING 8-OUNCE COTTON DUCK IN PURE CULTURE TESTS WITH AND WITHOUT GLUCOSE ADDED TO BASAL MINERAL SALTS—COTTON EXTRACT—AGAR MEDIUM. TWO WEEKS' EXPOSURE, 30°C, AGAR MAT TECHNIQUE

Fungi	Source	Percent change in strength: Mineral salts–cotton extract	Mineral salts–cotton extract–1% glucose
Pullularia sp. BPI 114	Awning, North Beach, Md.	+8	− 5
Pullularia sp. BPI 126	Fabric weathered above ground in Panama	− 2	−10
Pullularia sp. BPI 127	Fabric weathered above ground in Panama	− 1	− 1
Pullularia sp. BPI 128	Fabric weathered 12 mos. in New Orleans	+5	− 1
Pullularia sp. BPI 129	Wire cable insulation, weathered at Beltsville	− 1	+ 2
Pullularia sp. 1588	H. S. Sherwin	+7	− 3
Pullularia sp. A-1	L. M. Ames	0	+ 3
Pullularia sp. A-2	L. M. Ames	+5	0
Pullularia pullulans Weber F44	W. L. White	+8	+ 1
Pullularia pullulans J364	W. L. White	+6	+ 5
Pullularia pullulans PQMD 72h	W. L. White	+8	+ 3

TABLE VII. ACTIVITY OF SEVERAL ISOLATES OF SPECIES FROM THE ACTINOMYCETES IN TENDERING 8-OUNCE COTTON DUCK IN PURE CULTURE TESTS WITH AND WITHOUT GLUCOSE ADDED TO BASAL MINERAL SALTS MEDIUM. TWO WEEKS' EXPOSURE, 30°C, AGAR MAT TECHNIQUE. MEDIUM TO HEAVY GROWTH OCCURRED ON ALL SAMPLES IN THE PRESENCE OF GLUCOSE, WITH THE EXCEPTION OF #8664 (NO GROWTH) AND #8663 (VERY SLIGHT GROWTH)

Fungi	Source	Percent change in strength *Mineral salts only	Percent change in strength *Mineral salts–1% glucose
Nocardia erythropolis ATCC 4277	ATCC	+3	+6
Nocardia blackwellii ATCC 6846	ATCC	+6	+6
Streptomyces lipmanii ATCC 3331	ATCC	0	0
Streptomyces roseochromogenes ATCC 3347	ATCC	0	+3
Streptomyces scabies ATCC 3352	ATCC	0	+1
Streptomyces viridochromogenus ATCC 3356	ATCC	0	+2
Streptomyces albsonii ATCC 6852	ATCC	+3	+1
Streptomyces antibioticus ATCC 8663	ATCC	+2	−1
Streptomyces lavendulae ATCC 8664	ATCC	+3	+4
Streptomyces griseus ATCC 10137	ATCC	+2	+3

* Mineral salts medium suitable for growth of Actinomycetes—KH_2PO_4 2.04 grams/liter; K_2HPO_4 2.62 grams/liter; $NaNO_3$ 6.38 grams/liter; $MgSO_4 \cdot 7H_2O$ 2.20 grams/liter

breakdown. Of the 25 isolates within the group tested by White and coworkers [44], all were inactive. The species *A. janus* was not included in their tests.

A. terreus group. Ten isolates, representing 3 species, were all clearly active. All isolates produced a heavy, even growth on the fabric. White and coworkers [44] found 7 isolates within the species *A. terreus* to be active, but did not test *A. niveus* and *A. carneus.*

A. candidus group. No significant strength loss was observed with any of the 6 isolates of *A. candidus* which were tested. No other tests for cellulolytic activity as measured in terms of fabric breaking strength are known to have been made by earlier workers.

A. niger group. Of 13 isolates tested, activity was found in 2, both in the species *A. japonicus.* These results agree with recent observations by White and coworkers [46].

A. wentii group. Of 8 isolates, representing 3 species, none was found to be clearly active.

A. tamarii group. Of 8 isolates, representing 2 species, activity was found in a single isolate of *A. terricola.*

A. flavus-oryzae group. Of 11 isolates tested, none showed activity. This is in agreement with the data of White and coworkers [44], who found none of the several isolates which they tested within the group to be active.

A. ochraceus group. Of 10 isolates tested, representing 4 species, activity was found in 8 isolates, representing 3 of the 4 species.

Circinella. Data on this fungus, as well as on several of its close relatives in the group of the Mucoraceae—namely, *Cunninghamella, Syncephalastrum, Thamnidium,* and *Zygorhynchus*—are presented in the table. All isolates were found inactive. Further tests on isolates from the Mucoraceae were carried out in some detail and are described on pages 478–9.

Curvularia. It is interesting that all of the several fungi with dark-walled septate spores which were tested were active. Without claiming validity for any perfect generalization on the point, it can be noted, however, that activity was found in the genera *Curvularia, Alternaria, Coccosporium, Helminthosporium,* and *Diplodia. Stemphylium* [17] and *Brachysporium* [44] are known to be active from other work.

Fusarium. Of the 16 isolates of this important genus which were tested, all but 2 were active. If these data are added to the results of earlier workers [17, 44], one may conclude with some degree of certainty that the Fusaria are generally capable of decomposing cellulose. The species *F. moniliforme,* found very commonly on cotton weathered in the boll, is clearly active.

Hendersonia. The one isolate of this common

fabric-inhabiting fungus [48] which was tested was active.

Humicola. Under the described test conditions this fungus is an outstandingly rapid cellulose decomposer. Our results on this point check very closely with similar findings by White and coworkers [44], all of the 9 isolates tested by us and all of the 7 tested by them being highly active. *Monotospora dalcae* is a closely related form and, as may be seen from the table, is also very rapid in its action on cellulose.

Neurospora. This fungus, which has figured prominently in recent genetic and biochemical investigations, is clearly a cellulose decomposer. Because of its light color and fluffy growth, certain investigators might be inclined to class it intuitively as a "superficial" or non-cellulose-decomposing fungus. From the data, this is obviously not the case; many light-colored, fluffy fungi are actually highly cellulolytic.

Penicillium. Since fungi of this genus have been reported commonly on cotton in moist or damp storage [23], data on cellulose-decomposing activity within the genus are of interest. In the most recent contribution on the subject—namely, that by White and coworkers [44]—determinations were carried out on 94 cultures, representing 62 named species and 32 unknown species. The only clearly positive results obtained were for certain of the 9 strains representing the *P. lutcum* series, for 4 of the 20 strains of the *P. citrinum* series, and for 2 isolates of unknown species. Strains reported as *P. lutcum* had, in fact, been found active in previous work by Thom, Humfeld, and Hollman [38], and by Klemme and coworkers [17]. Tests by Scales [33] had indicated a rather broad distribution of activity throughout the genus, but his results have been questioned by later workers. Otto [28] found definite activity in two Penicillia but gives no indication of species. Heyes and Holden [11] found strong activity with *P. purpurogenum* var. *rubri sclerotium* and with *P. pinophilum.*

In our own work (Table II) we have found activity not only in the *P. lutcum* series, 8 active isolates having been found among the 16 isolates tested, but also in certain isolates from 14 other series—namely, the *javanicum* (3 species), *thomii* (2 species), *implicatum* (1 species), Ramigena (1 species), *raistrickii* (1 species), *janthinellum* (3 species), *canescens* (1 species), *citrinum* (2 species), *oxalicum* (1 species), *brevi-compactum* (1 species), *commune* (1 species), *urticae* (1 species), *funiculosum* (2 species), and *purpurogenum* (2 species) series. The appearance of the growth of 2 of these isolates on the fabric strips is shown in Figure 1.

Phoma. Two out of the 3 isolates tested were active.

Pullularia pullulans. Tests on 3 isolates obtained from Dr. W. L. White all showed activity to be lacking, which is in agreement with his results [44]. Results of further tests are presented in Table VI.

Sordaria. This fungus not only caused rapid breakdown but also made an unusually smooth, heavy growth on the fabric.

Stachybotrys. Results on the 3 isolates of this fungus which were tested agree with all previous findings on *Stachybotrys* and the closely related *Memnoniella* in indicating activity to be general with these forms [9, 17, 22, 44].

Thielavia. Both isolates were active, produced a thick, smooth mycelial mat on the fabric, and sporulated in abundance.

Table III presents results showing the effect of temperature on the breakdown of cotton fabric by *Cladosporium herbarum.* This fungus is of very common occurrence on cotton weathered in the field before harvest [23], as well as on weathered cotton fabric and on dew-retting flax and hemp [23], and consequently is of great interest from a practical standpoint. Klemme and coworkers [17] tested 1 isolate of *Cladosporium* and 4 of the closely related *Hormodendrum* and found all isolates to be significantly but, in general, only moderately active. White and coworkers [44] tested 22 isolates of *Cladosporium* and found only 12 of them to be significantly active; of the active isolates, none showed very rapid or severe fabric breakdown. Basu [1] found one isolate to be inactive.

It seemed possible, in view of the fact that *Cladosporium* isolates had been reported earlier to grow well at low temperatures [2, 15], that the temperature of approximately 30°C (86°F) used by Klemme and coworkers [17], by White and coworkers [44], and by Basu [1] was too high for optimum growth, or even any growth, of some isolates of this fungus. When tests were carried out under the same conditions as have been specified for the data of Table II but at a series of different temperatures, the results shown in Table III were obtained. It is clear that 30°C is, in general, too high a temperature for this

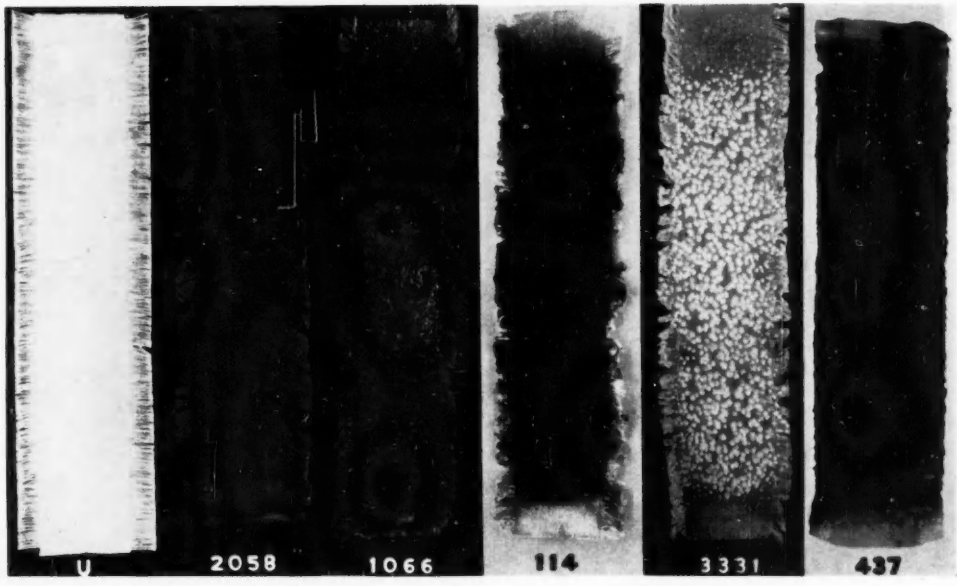

FIG. 1. *Test strips before and after growth of fungi.*

U = *original uninoculated strip.*
2058 = *strip after 2-week growth of* Penicillium multicolor *NRRL 2058 in the presence of mineral salts only, — 90% change in strength.*
1066 = *strip after 2-week growth of* Penicillium purpurogenum *var.* rubri-sclerotium *NRRL 1066 in the presence of mineral salts only, — 96% change in strength.*
114 = *strip after 2-week growth of* Pullularia pullulans *BPI 114 in the presence of glucose, cotton extract, and mineral salts, — 5% change in strength.*
3331 = *strip after 2-week growth of* Streptomyces lipmanii *ATCC 3331 in the presence of glucose and mineral salts, 0% change in strength.*
437 = *strip after 2-week growth of* Rhizopus microsporus *Cutter's 437 in the presence of glucose and mineral salts; — 3% change in strength.*

fungus, since only 3 of the 10 isolates showed definite activity after 2 weeks in our tests at 30°C, while all 10 were active at 20°C.

Table IV presents data from experiments carried out on certain of the Aspergilli on which the results of tests have already been reported for a mineral salts agar procedure in Table II. It was desired to ascertain whether the lack of growth and of fabric breakdown observed with certain of the Aspergilli as reported in Table II was definitely due to lack of cellulolytic activity on the part of the fungi concerned or merely to inadequacy of the mineral salts medium used for their growth. It is clearly apparent from the data of Table IV that the mineral salt composition of the medium was satisfactory, since heavy growth was obtained in all cases when glucose was added to it. This heavy growth was not accompanied, however, by fabric tendering except in cases where the fungi had already been found to be active on the mineral salts medium (without glucose) of Table II.

Table V is concerned with a group of 18 isolates of Phycomycetes belonging to the Mucoraceae. Tests on 7 isolates of this same group have already been presented in Table II, with none of the forms tested showing activity. These results are in general agreement with limited previous findings by other workers [12, 28, 29, 40, 44]. These earlier workers, however, have not claimed that the basal growth medium used in their experiments was adequate for the growth of these fungi, nor is information on this matter available from our own data of Table II.

Although Paine's [29] results have been widely quoted, he notes in his paper the possibility that the growth medium used may have been unsuited to the mucoraceous fungi tested. White and coworkers [44] used bleached sheeting in their tests, a somewhat questionable procedure in view of the possibility of vitamin requirements of the fungi under test.

When glucose was added to our basal salts medium (p. 464) and several mucoraceous fungi were tested on gray cotton duck ("Q" duck), all isolates grew luxuriantly. Parallel tests were carried out in the absence of glucose. In no case was a significant strength loss observed. A much stronger case, therefore, can now be presented from the data of Table V to prove that the Mucoraceae are unable to decompose cellulose. A test strip showing heavy growth of a *Rhizopus* in the presence of glucose is seen in Figure 1.

Table VI presents data on isolates of *Pullularia pullulans* from experiments similar in purpose and principle to those of Tables IV and V. Medium-to-heavy growth occurred in all cases in the presence of added glucose (Figure 1) but no significant strength losses occurred in any of the tests.

Table VII is concerned with tests on 10 isolates of actinomycetes. Because of the known preference of these fungi for a somewhat more alkaline medium than is best for other forms, the salt medium was slightly altered. Strength losses were insignificant in all cases, in spite of the generally medium-to-heavy growth obtained with the glucose-containing substrate. A strip with heavy growth from a glucose-containing culture is shown in Figure 1. These data, in conjunction with present lack of detailed evidence indicating actinomycetes to be a major component of the fungus flora of deteriorating cellulose fiber [23], suggest that the actinomycetes are probably not of great importance in the deterioration of such fibers.

Discussion

It was mentioned in the Introduction that a number of papers other than those cited there contain information on which fungi can and which cannot decompose cellulose. In numerous cases the nature of the experimental methods reported in these papers renders it difficult to arrive at many definite conclusions from the data presented. It appears desirable for certain reasons, however, to attempt a rather critical review of them. Many unwarranted original conclusions have been accepted in an uncritical manner in textbooks and reported in other general presentations but no critical and complete review has been attempted. In general, it appears to be true that data obtained by careful application of sound methods are in good accord with the data presented here and by others employing the fabric strip-culture bottle method, as are also the *more striking results* obtained by non-quantitative observational methods. Data in conflict with those obtained in fabric tests are generally of a doubtful character. These statements should not, of course, be accepted without adequate evidence, but if they can be substantiated they should lead to the elimination of some of the confusion existent in the literature on the subject.

The earliest paper presenting experimental evidence on cellulose-decomposition by the mold fungi appears to be a report by van Iterson [12], published in 1904. Van Iterson isolated several fungi on filter paper in the presence of a mineral salts solution, identified 15 of them, and re-inoculated these 15 in pure culture upon filter paper in the presence of mineral salts. Although few details are given, the degree of cellulose-decomposing activity apparently was judged primarily on the basis of the extent of growth of the fungus, the microscopic appearance of the fiber, and its loss in weight. Most of the fungi tested have been investigated more thoroughly by more recent workers. Certain points in the paper are of interest, however—notably, his finding of activity in a *Chaetomium* species and his failure to find it in a *Mucor* and in *Dematium pullulans* (*Pullularia pullulans*). Each of these observations is in accord with our own data and with the findings of other later workers. Even more interesting is his singling out of "*Mycogone puccinioides*" as the most active of the 15 cultures tested by him. According to Diehl [3], van Iterson's drawing of his "*Mycogone puccinioides*" indicates that it was in reality the same as or very closely related to the *Humicola* of other workers. Numerous isolates of *Humicola* were found to be extremely rapid in their action on cellulose by White and coworkers [44] and by ourselves in the present study.

A short section from a paper by Krainsky [18] published in 1914 has been cited by numerous more recent writers as evidence indicating that the actinomycetes generally are capable of decomposing cellulose. The original observations, however, seem decidedly inadequate as a basis for any generalizations

on the point, and the author's statement ([18], page 674) to the effect that most actinomycetes grow well on filter paper must be interpreted with caution. In most cases no microscopic examinations or quantitative determinations which indicated severe fiber disintegration appear to have been made. A few cultures apparently were definitely active. Waksman [41] states: "When compared with the bacteria and fungi capable of decomposing cellulose, the action of most of the actinomyces upon cellulose is only slow and limited." Apparently he regarded the high cellulolytic activity recorded in a 1931 paper [40] for "Actinomyces 389, closely related to *Act. violaceus ruber*, a typical soil *Actinomyces*," as being in some way aberrant or unusual. Dubos [4], Meyer [25], and Jensen [16] likewise reported finding single active actinomycete isolates. Lieske ([20], page 150) apparently tested numerous isolates of actinomycetes on filter paper but obtained such very slight growth that it was impossible for him to conclude that any of his isolates were certainly cellulose decomposers.

In a much-cited paper published in 1915, Scales [33] reported that he tested 20 named species of *Penicillium* for cellulose-decomposing activity on reprecipitated-cellulose agar and found 15 of them to produce a cleared zone in the agar around the colony, this phenomenon being taken to indicate the presence of cellulolytic activity. Scales' results are quite in contrast to those of White and coworkers [44], who tested a large number of isolates of *Penicillium* on cotton fabric and found activity only in members of the *P. luteum* series, in *P. citrinum*, and in 2 isolates unidentified to species. Scales [33] also tested 8 Aspergilli by the reprecipitated-cellulose agar method; of these he found all except *A. candidus* to be active, positive results being recorded even for *A. flavus*, *A. oryzae*, and *A. wentii*, forms found by White and coworkers [44] and by ourselves in the present work to give negative results in fabric tests. Experiments demonstrating the ability of organisms to produce clearing of a reprecipitated-cellulose agar have frequently not been accompanied by data indicating also their ability to decompose native undegraded cellulose, such as occurs in a cotton fiber. The work of Scales on fungi [33], in common with some of the work involving the use of similar techniques with bacteria [5, 24], can, therefore, probably be correctly regarded as of doubtful significance with respect to problems of fiber deterioration.

In 1916 Otto [28] presented the results of a thorough study of the action of pure cultures of several fungi on various types of cellulosic fibers—notably, different types of filter paper, parchment paper, cotton fiber, flax fibers, and basswood fibers. His tests were carried out in the presence of mineral salts solutions, both with and without added glucose, and the fibers were subjected to careful microscopic examination after periods of a few to several weeks. Convincing evidence is offered to indicate cellulolytic activity for *Stemphylium*, "*Mycogone*," *Stachybotrys*, *Trichoderma*, *Botrytis*, one of 2 *Cladosporium* isolates, and 2 *Penicillium* isolates, with activity being absent in one *Cladosporium*, one *Penicillium*, an *Aspergillus niger*, 4 *Mucors*, and a *Rhizopus*. These data, while limited, are in excellent agreement with our own and with those of other workers using the cotton fabric test method. There appears to be reason to believe that Otto's "*Mycogone*," like that of Iterson [12] and of other authors [8, 13, 14], may have been the same as or very similar to the fungus recorded here and by others under the generic name of *Humicola* [4, 19, 40, 44].

Paine [29] inoculated pure cultures of several fungi upon cotton and filter paper in the presence of mineral salts and made a record of the occurrence or non-occurrence of visible fungus growth. Such use of visual estimates of growth with the naked eye as a sole criterion of cellulolytic activity without supporting quantitative data has been regarded generally by more recent workers as a doubtful basis for conclusions, especially when anything less than luxuriant growth and severe fiber disintegration occurs. An amount of fungus growth which if actually weighed would be found to be extremely small can in some cases look deceptively great and can take place on very small amounts of impurities in the system, while, conversely, what appears to the naked eye to be a relatively small or an insignificant amount of growth could conceivably cause a very significant loss in fiber strength. Paine's results are therefore probably of little significance, and the fact that they do not agree in detail with later findings should cause little concern. One of his conclusions, however, has been substantiated in part. On the basis of tests of a total of 7 isolates from 4 genera in the Mucoraceae, Paine came to the tentative conclusion that—"It would appear that the Phycomycetes are not active fermenters of cellulose or the medium used was not suitable for their growth." The Mucoraceae actually

constitute only a part of the Phycomycetes and the generalization is too broad.

Galloway [6] observed the amount of visible growth of several fungi on filter paper in the presence of mineral salts. His experiments suffer from the same uncertainties of interpretation mentioned above in the case of work by Paine [29]. It is interesting to note, however, that his ratings of "strong" for *A. fumigatus*, *A. nidulans*, *A. terreus*, and *A. flavipes*, of "nil" for *A. candidus*, and also of "good activity" for *A. ustus* and *A. ochraceus* by a similar method in a later paper [7] agree with our own findings in the present paper. From results presented here it appears that his intermediate ratings of "moderate" for others of the Aspergilli are unreliable and may have been due in several cases to growth of the fungus on non-cellulosic material carried over in the inoculum or on other non-cellulosic impurities in the system.

Norman [27] subjected oat straw to the action of pure cultures of several fungi for 48-day periods; he found major decreases in cellulose content to occur with each of several of the forms tested, including *Aspergillus versicolor*, 6 isolates of which gave no strength losses of cotton fabric in the experiments of White *et al.* [44], and *Aspergillus niger*, numerous cultures of which were also inactive in White's experiments. Results reported in the present paper agree with those of White and coworkers [44] rather than with those of Norman, although *A. janus* in the *A. versicolor* group was clearly active (Table II).

Waksman reported [40] a chemical-analytical study of the decomposition of wheat and rye straw and oak leaves by fungi. *Trichoderma* decomposed cellulose in all three cases and *Zygorhynchus* (one of the Mucoraceae) failed to decompose cellulose in the two cases tested—namely, the oak leaves and the rye straw—while *Humicola* was active against the same two materials. These findings are in good accord with more recent fabric-culture bottle test results.

Heyes and Holden [11] stated that tendering of cotton could be brought about by *Penicillium purpurogenum* var. *rubri sclerotium* and by *Penicillium pinophilum*. The former of these demonstrated strong cellulolytic activity in the tests of Table II. Its appearance on the strips may be seen in Figure 1.

Niethammer [26] suggested that the species of Penicillia falling under the subdivision Asymmetrica be divided into six subgroups and those under the Symmetrica into two subgroups. Weak growth on filter paper but no ability to actually decompose cellulose was stated as characteristic of the *P. luteum* and *P. purpurogenum* subgroups, and weak or no growth on cellulose was reported for each of the subgroups in the Asymmetrica except that represented by *P. italicum*. It is not clear from the account that anything more than a rather superficial testing of a few isolates was actually carried out and the generalizations made are not borne out by the data here presented.

Whiffen [43] demonstrated cellulose decomposition by 7 species of chytrids (primitive aquatic Phycomycetes) by the method of weight loss of filter paper. She made the interesting and apparently correct statement that ". . . previously no representative of the Phycomycetes has been found capable of decomposing cellulose. . . ." Stanier [36] and Quantz [31] both confirmed the fact that the chytrid *Rhizophlyctis rosea* utilizes filter paper as a source of carbon.

Basu [1] has recently described experiments in which several fungi were tested for their ability to grow on filter paper in the presence of mineral salts. He used 11 named species of Aspergilli, 19 unnamed Penicillia, and a variety of other fungi, mostly unidentified. Of the 69 isolates tested, 33 appeared to grow strongly on filter paper as a source of carbon. The author notes the difficulty of interpreting his own data from these experiments in the following words: "It was obvious, however, that the value of such data was limited, because visual impression of the amount of growth may often be a misleading index of the degree of decomposition." He might have added that in numerous borderline cases it is difficult to determine by such methods whether the fungi are actually active in decomposing cellulose at all. When cellulose determinations were carried out on filter paper after a period of incubation with several different fungi, losses of from 6 to 71 percent occurred. *Aspergillus sydowi* caused a 50-percent loss in cellulose, an interesting finding in view of the fact that White and coworkers [44] tested a large number of isolates of this fungus on cotton fabric without being able to obtain any breakdown.

In the examination of tendered fibers to diagnose the cause of deterioration, it is frequently desirable to know whether a particular fungus is potentially

capable of decomposing cellulose or not. It is hoped that the original and collected information presented here may be of value in this connection. The mere observation and identification on a tendered sample of a fungus known to be cellulolytic under experimental conditions does not prove, of course, that fiber tendering has taken place as a result of fungus action. The presence of an abundance of fungus growth, the localization of fiber tendering at the sites of greatest growth [48], and evidence indicating that greatest tendering takes place under conditions known to be conducive to the growth of fungi—these are all important in the diagnosis. Numerous tests not here reported in the tables but carried out according to methods exactly similar to those employed for Table VI have shown that the yeast *Rhodotorula aurea*, which occurs on field-deteriorated cotton, is unable to tender cotton fabric, even though it grows very well in contact with the fabric test specimens in the cultures containing glucose. Many excellent photomicrographs of the mold fungi are available in a book by G. Smith [35], which may be valuable as an aid in fungus identification as well as for the excellent information it contains on practical methods of working with the fungi in the laboratory.

It is interesting to note that cellulolytic activity appears to be, at least in many cases, a rather permanent property of a fungus which is not readily lost by long-continued propagation on non-cellulosic media. With few exceptions, the fungi tested for cellulolytic activity in the present work had been isolated and grown, many of them for periods of several years, on non-cellulosic media. Tests of *Chaetomium globosum* USDA 1042.4 after twelve successive transfers on a high-sugar medium failed to indicate that this treatment had brought about any measurable loss of cellulolytic activity. Isolates of *Humicola* are notably subject to sectoring—*i.e.*, mutation—and yet, in spite of this, have shown uniformly high cellulolytic activity in all tests. *Helminthosporium* isolates are especially noted for their habit of losing the power of sporulation. All 10 of the *Helminthosporium* isolates tested by us, however, show cellulolytic activity, whereas it cannot be proven from the data here presented that some, or even many, of the numerous *Penicillium* isolates found inactive in our tests were not originally cellulolytic. We have no reason to suspect that this really is the case.

Among the fungi, the ability to utilize cellulose as a source of carbon and energy seems to be much more widespread than the ability to utilize certain other naturally occurring substances. The authors are not aware, for example, of presently available evidence to indicate that wool can be utilized by any except a small group of specialized genera among the fungi, unless the fiber is subjected first to a partial degradation by moist heat or some other agent. This situation is perhaps not an unexpected one since cellulose exists in much larger amounts in nature than does wool, as, for example, in the most common habitat of the fungi—the soil. Lignin, very widespread in nature, is also thought, however, to be utilized only by a relatively specialized group of fungi [42]. The hemicelluloses, on the other hand, are susceptible to attack by many fungi [37]. Articles made of leather, rubber, and cork were treated with mildew preventives during World War II, but it was generally agreed that it was the various additives, conditioning agents, and binders present which supported most or all of the growth and made a protective agent desirable. Fibers made of nylon and Vinyon are highly resistant if not immune to fungus attack [21], as are also cellulose acetate [21] and many other cellulose derivatives [34]. While a few bacteria have been demonstrated to decompose native cellulose in pure culture, all present indications point to a much wider distribution of this faculty among the fungi than among the bacteria.

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Literature Cited

1. Basu, S. N., Fungal Decomposition of Jute Fiber and Cellulose: I: Preliminary Survey of Commonly Occurring Species; II: Effect of Some

Environmental Factors, *J. Text. Inst.* **39**, T232–7, T237–48 (1948).

2. Brooks, F. T., and Hansford, C. G., Mould Growths Upon Coldstore Meat, *Trans. Brit. Myc. Soc.* **8**, 113–42 (1922).
3. Diehl, W. W., Identity of Iterson's Cellulolytic Mycogone, *Mycologia* (in press).
4. Dubos, R. J., Decomposition of Cellulose by Aerobic Bacteria, *J. Bact.* **15**, 223–34 (1928).
5. Fuller, W. H., and Norman, A. G., A Cellulose-Dextrin Medium for Identifying Cellulose Organisms in Soil, *Proc. Soil Sci. Soc. Am.* **7**, 242–6 (1942).
6. Galloway, L. D., Fungi Causing Mildew in Cotton Goods. *J. Text. Inst.* **21**, T277–86 (1930).
7. Galloway, L. D., Indian Soil Fungi, *Indian J. Agr. Sci.* **6**, 578–85 (1936).
8. Golebiowska, J., Przyczynek do badan nad rozkladem blonnika przez grzyby nizsze wystepujace w glebie (Contribution to the Study of Cellulose Decomposition by Soil Moulds), *Ann. Univ. Mariae Curie-Sklodowska* **2E**, 223–48 (1947). Abstract in *Soils and Fertilizers* **11**, 313 (1948).
9. Greathouse, G. A., Klemme, D. E., and Barker, H. D., Determining the Deterioration of Cellulose Caused by Fungi, *Ind. Eng. Chem., Anal. Ed.* **14**, 614–20 (1942).
10. Greathouse, G. A., and Ames, L. M., Fabric Deterioration by Thirteen Described and Three New Species of *Chaetomium*, *Mycologia* **37**, 138–55 (1945). Abridged in TEXTILE RESEARCH JOURNAL **15**, 223–5 (June 1945).
11. Heyes, T. F., and Holden, H. S., Investigation of the Action of Certain Species of *Penicillium* on Artificial Silk, *J. Text. Inst.* **23**, T79–94 (1932).
12. Iterson, C. van, Die Zersetzung von Cellulose durch aerobe Mikroorganismen, *Centr. Bakt. Parasitenk.* **11**, 689–98 (1904).
13. Jensen, C. N., Fungous Flora of the Soil. *Cornell Agr. Exp. Sta. Bull.* **315**, 415–501 (1912).
14. Jensen, H. L., Microbiology of Farmyard Manure Decomposition, II: Decomposition of Cellulose, *J. Agr. Sci.* **21**, 81–100 (1931).
15. Jensen, H. L., Microorganisms Active in the Dew-Retting of Flax, *Australian J. Sci.* **4**, 59 (1941).
16. Jensen, H. L., Observations on Properties of Certain Fungicidal Compounds, *Proc. Linnean Soc. N. S. Wales* **71**, 119–29 (1946).
17. Klemme, D. E., Greathouse, G. A., Bollenbacher, K., and Pope, S., "Deterioration of Cotton Fabric by Certain Microorganisms," *U. S. Dept. Agr. Circ. 437*, 1945, pp. 1–11.
18. Krainsky, A., Die Aktinomyceten und ihre Bedeutung in der Natur, *Centr. Bakt. Parasitenk.* **41**, 649–88 (1914).
19. Kubiena, W., and Renn, C. E., Micropedological Studies of the Influence of Different Organic Compounds Upon the Microflora of the Soil, *Zentr. Bakt. Parasitenk.*, Abt. 2, **91**, 267–92 (1935).
20. Lieske, R., "Morphologie und Biologie der Strahlenpilze," Leipzig, Germany, Borntraeger, 1921, pp. 1–292.
21. Marsh, P. B., Mildew- and Rot-Resistance of Textiles, TEXTILE RESEARCH JOURNAL **17**, 597–615 (Nov. 1947).
22. Marsh, P. B., and Bollenbacher, K., Vitamin Requirements of *Memnoniella* and *Stachybotrys*, *Am. J. Bot.* **33**, 245–9 (1946).
23. Marsh, P. B., and Bollenbacher, K., Fungi Concerned in Fiber Deterioration, I: Their Occurrence, TEXTILE RESEARCH JOURNAL **19**, 313–24 (June 1949).
24. McBeth, I. G., and Scales, F. M., "Destruction of Cellulose by Bacteria and Filamentous Fungi," *U. S. Dept. Agr., Bur. Plant Industry Bull. 266*, pp. 1–52 (1913).
25. Meyer, R., Beitrage zur Kenntnis der Cellulosezersetzung unter niedriger Sauerstoffspannung, *Arch. Mikrobiol.* **5**, 185–222 (1934).
26. Niethammer, A., Die Gattung Penicillium, *Zentr. Bakt. Parasitenk.*, Abt. 2, **98**, 65–76 (1938).
27. Norman, A. G., Biological Decomposition of Plant Materials, Part IV: Biochemical Activities on Straws of Some Cellulose-Decomposing Fungi, *Ann. Applied Biol.* **18**, 244–59 (1931).
28. Otto, H., Untersuchungen über die Auflosung von Zellulosen und Zellwanden durch Pilze, *Beitr. allgemeinen Botan.* **1**, 190–260 1916).
29. Paine, F. S., Studies of the Fungous Flora of Virgin Soils, *Mycologia* **19**, 248–67 (1927).
30. Pope, S., A New Species of *Metarrhizium* Active in Decomposing Cellulose, *Mycologia* **36**, 343–50 (1944).
31. Quantz, Ludwig, Untersuchungen über die Ernahrungsphysiologie einiger niederen Phycomyceten, *Jahrb. wiss. Botan.* **91**, 120–68 (1943).
32. Raper, K. B., and Thom, Chas., "A Manual of the Penicillia," Baltimore, Maryland, Williams and Wilkins Co. (in press).
33. Scales, F. M., Some Filamentous Fungi Tested for Cellulose Destroying Power, *Botan. Gaz.* **60**, 149–53 (1915).
34. Siu, R. G. H., and White, W. L., "Microbiological Degradation of Cotton Fabrics," U. S. Office of The Quartermaster General, Research and Development Branch, Microbiological Series Report No. 1 (released for public information by the Office of the Publication Board, U. S. Dept. of Commerce, 1945), pp. 1–64.
35. Smith, G., "An Introduction to Industrial Mycology," London, Edward Arnold and Co. Ltd., 1946, pp. 1–271.
36. Stanier, R. Y., Cultivation and Nutrient Requirements of a Chytridiaceous Fungus, *Rhizophlyctis rosea*, *J. Bact.* **43**, 499–520 (1942).
37. Thaysen, A. C., and Bunker, H. J., "Microbiology of Cellulose, Hemi-Celluloses, Pectin and Gums," London, Oxford University Press, 1927, pp. 1–363.

38. Thom, C., Humfeld, H., and Holman, H. P., Laboratory Tests for Mildew-Resistance of Outdoor Cotton Fabrics, *Am. Dyestuff Reptr.* **23**, 581–6 (1934).
39. Thom, C., and Raper, K. B., "A Manual of the Aspergilli," Baltimore, Maryland, Williams and Wilkins Co., 1945, 76 figures, pp. 1–373.
40. Waksman, S. A., Decomposition of the Various Chemical Constituents, etc., of Complex Plant Materials by Pure Cultures of Fungi and Bacteria, *Arch. Mikrobiol.* **2**, 136–54 (1931).
41. Waksman, S. A., "Principles of Soil Microbiology," Second Edition, Baltimore, Maryland, Williams and Wilkins Co., 1932, pp. 1–894.
42. Waksman, S. A., Decomposition of Lignin, *in* "Wood Chemistry," edited by L. E. Wise, New York, Reinhold Publishing Corp., 1944, pp. 1–900.
43. Whiffen, Alma J., Cellulose Decomposition by the Saprophytic Chytrids, *J. Elisha Mitchell Sci. Soc.* **56**, 321–9 (1940–1941).
44. White, W. L., Darby, R. T., Stechert, G. M., and Sanderson, K., Assay of Cellulolytic Activity of Molds Isolated from Fabrics and Related Items Exposed in the Tropics, *Mycologia* **40**, 34–84 (1948).
45. White, W. L., and Downing, M. H., Identity of "*Metarrhizium glutinosum*," *Mycologia* **39**, 546–55 (1947).
46. White, W. L., Siu, R. G. H., and Reese, E. T., The Black Aspergilli in Relation to Cellulosic Substrata, *Bull. Torrey Botan. Club* **75**, 604–32 (1948).
47. Zuck, R. K., Isolates Intermediate Between *Stachybotrys* (Corda) and *Memnoniella* (von Hohnel), *Mycologia* **38**, 69–76 (1946).
48. Zuck, R. K., and Diehl, W. W., On Fungal Damage to Sun-Exposed Cotton Duck, *Am. J. Botany* **33**, 374–82 (1946).

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Specificity of Microbiological Attack on Cellulose Derivatives

R. G. H. Siu, Richard T. Darby, Paul R. Burkholder, and E. S. Barghoorn

Contribution of the Quartermaster Research and Development Laboratories, Philadelphia, Pennsylvania; the Osborn Botanical Laboratories, Yale University, New Haven, Connecticut; and the Biological Laboratories, Harvard University, Cambridge, Massachusetts

Abstract

Data have been presented relating to the growth of 11 species of microorganisms isolated from deteriorated cotton fabrics on various derivatives of glucose, mannose, cellobiose, and cellulose. A high degree of specificity was exhibited. As long as there was at least one firmly bound substituent in every anhydroglucose unit, the resulting derivative was not susceptible to microbiological attack. Under such conditions the nature of the substituent has relatively little influence on the degree of resistance imparted. This was considered to be promising theoretical support for the underlying premise on which the idea of multipurpose topochemical reactions as a means of mildewproofing cotton fabrics was proposed.

SINCE DORÉE'S DISCOVERY in 1920 [2] of the resistance of cellulose acetate to sea water much work has been done on the topochemical acetylation of cellulosic fibers as a means of mildewproofing. Thaysen and associates [8, 9] in England were among the pioneers in this field. Investigations were continued in this country, chiefly at the Southern Regional Research Laboratory [3]. During recent years methods of imparting mildew-resistance by topochemical reactions have been expanded [7] into

TABLE I. GROWTH OF FUNGI ON SUGAR DERIVATIVES

Sugar derivatives*	*Aspergillus niger* PQD 72A	*Aspergillus flavipes* Fla. A14	*Pestalotia* sp. PQD 79C	*Chaetomium globosum* USDA 1042.4	*Gliocladium* sp. PQD 3A	*Penicillium* sp. USDA 15.1	*Memnoniella echinata* PQD 1C	*Gliomastix convoluta* PQD 4C	*Myrothecium verrucaria* USDA 1334.2
Glucose	+	+	+	+	+	+	+	+	+
3-Methyl-α-*D*-glucose	?	−	−	−	−	?	−	?	−
2,3,6-Triethyl-*D*-glucose	−	−	−	−	−	?	−	−	−
Tetramethylglucopyranose	−	−	−	−	−	−	−	−	−
Pentacetyl-α-*D*-glucose	−	−	?	−	−	−	−	−	−
Pentacetyl-β-*D*-glucose	−	−	?	−	−	−	−	−	−
Glucose phenylosazone	−	−	−	−	−	−	−	−	−
Methyl-α-*D*-glucoside	?	−	−	?	?	+	−	+	−
Methyl-β-*D*-glucoside	+	+	+	+	+	+	+	+	+
Methyl-2,3,6-triethyl-β-*D*-glucoside	−	−	−	−	−	−	−	−	−
Mannose	+	+	+	+	+	+	+	+	+
4-Methyl-*D*-mannose	−	−	−	−	−	?	−	−	−
Methyl-α-*D*-mannoside	−	−	−	?	−	?	−	?	−
Methyl-4-methyl-α-*D*-mannoside	−	−	−	?	−	?	−	−	−
Cellobiose	+	+	+	+	+	+	+	+	+
Cellobiose heptacetate	−	−	−	−	−	−	−	−	−
Cellobiose octacetate	−	−	−	−	−	−	−	−	−
Heptacetyl ethylcellobioside	−	−	−	−	−	−	−	−	−

* Appreciation is expressed to Drs. C. S. Hudson, M. L. Wolfrom, and R. S. Bower for these compounds.

a general approach to the problem of simultaneously, and by a single chemical reaction, rendering cellulose mildew-resistant and flameproof, repellent to water, and/or resistant to light. These attempts have been based on the premise that within broad limits the nature of the chemical substituent does not itself fundamentally determine the degree of mildew-resistance. Such resistance *per se* is imparted to the cellulose molecule by chemical substituents bound to the molecule in the proper position and numbers. Accordingly, it seems possible to select substituents which, because of their inherent nature, impart other desirable properties to the fabric. The present paper presents data in support of these generalizations. It also attempts to elucidate further the mechanism of breakdown of cellulose molecules by microorganisms.

TABLE II. GROWTH OF *Myrothecium verrucaria* ON CELLULOSE DERIVATIVES

Preparation	Source*	Description	% weight loss†	Growth rating
Filter paper	Whatman	Ground to 60 mesh	69	+++
Oxidized cellulose	E. K.	Prepared with nitrogen oxide	11	+
Methyl cellulose	Dow	D.S. > 1.0	8	0—+
Methyl cellulose	Dow	D.S. = 2.0	8	+
Ethyl cellulose	Dow	D.S. = 2.25 to 2.58	0	0
Cellulose acetate	S. R. R. L.	D.S. = 1.0; 22.3% AcO	0	0
Cellulose triacetate	E. K.	—	0	0
Cellulose acetate butyrate	E. K.	16% butyryl	0	0
Cellulose acetate hydrogen phthalate	E. K.	—	8	0
Cellulose acetate stearate	E. K.	—	1	0—+
Tosyl cellulose	I. P. C.	D.S. = 1.18 to 2.01	0	0—+
Iodotosyl cellulose	I. P. C.	D.S. = (0.60 to 0.86 iodotosyl (0.58 to 1.15 tosyl	2	0—+
Carboxymethyl cellulose	S. R. R. L.	D.S. = 0.067	56	++
Carboxymethyl cellulose, sodium salt	S. R. R. L.	D.S. = 0.2	>66	++
Carboxymethyl cellulose, aluminum salt	S. R. R. L.	D.S. = 0.067	>56	+

* E.K. = Eastman Kodak. S.R.R.L. = U.S.D.A. Southern Regional Research Laboratory. I.P.C. = Institute of Paper Chemistry. The authors are grateful to these sources for the compounds listed.

† Each value based on three or more flasks.

Experimental Methods and Results

1. Growth of Fungi on Sugar Derivatives

Qualitative observations were made on the growth of 9 species of fungi on sugar derivatives. Ten mg. of each of the compounds listed in Table I were placed in 50-ml. Erlenmeyer flasks and autoclaved at 120°C for 20 minutes. To the separate flasks there was added 1 ml. of a spore suspension from each of the various organisms. Two replicate cultures were incubated at 30°C for 3 days and then examined for mycelial growth. The results are given in Table I, with "+" indicating definite growth, "−" no growth, and "?" little or questionable growth.

2. Growth of Myrothecium verrucaria *on Cellulose Derivatives*

Studies were carried out on the susceptibility of various preparations of cellulose derivatives to attack by the active cellulose-degrading fungus, *Myrothecium verrucaria*. The shaker-flask method [5, 6] was used. One hundred mg. of the sample was placed with 20 ml. of a mineral salts solution in a 125-ml. Erlenmeyer flask. The salt solution was prepared with the following concentrations: $MgSO_4$ 0.009*M*, KH_2PO_4 0.02*M*, K_2HPO_4 0.012*M*, and NH_4NO_3 0.0375*M*. The final mixture of solution and cellulose derivatives was adjusted to pH 6.5 by 0.1*N* KOH. After sterilization by autoclaving, each flask, after cooling, was inoculated with 1 ml. of a pregerminated spore suspension and incubated on the shaker at 30°C for 9–10 days. The loss in dry weight of the substrate and the visual growth ratings were taken as indices of susceptibility. The data are given in Table II.

3. Effect of Degree of Polymerization and Degree of Substitution

Similar tests were carried out with cyanoethylcellulose preparations of various degrees of substitution. These were prepared by the reaction of acrylonitrile on cotton. Data on the susceptibility of the different samples are presented in Table III.

TABLE III. UTILIZATION OF CYANOETHYLCELLULOSE* PREPARATIONS BY *Myrothecium verrucaria*

Sample No.	% N	No. of substituents per anhydroglucose unit	% weight loss in 13 days
1	0	0	80
2	6.64	1.02	29
3	8.43	1.43	6
4	9.98	1.85	2
5	10.50	2.02	4
6	11.44	2.35	0
7	12.87	2.9	0

* Gratitude is expressed to Rohm and Haas Co. of Philadelphia for this series of preparations.

In the next series 4 species of fungi and one of bacteria (*Sporocytophaga myxococcoides* USDA 482), which had been isolated from deteriorating cotton, were used. Cellulose acetates with varying degrees of substitution and polymerization were employed as sole sources of carbon for the organisms. The acetates were prepared by Dr. E. Heuser of the Institute of Paper Chemistry by the acetylation of cellulose in phosphoric acid solutions [4]. The acetate with a low degree of substitution and a low degree of polymerization was prepared by acetylating dissolved cotton linters in 100-percent phosphoric acid with a limited quantity of acetic anhydride without cooling for about 1½ hours, followed by precipitation with diethyl ether. By means of different combinations of temperature and duration of acetylation, he succeeded in obtaining highly or lowly substituted acetates with either a high or a low degree of polymerization. Inasmuch as the reaction occurred in solution, it is assumed that partial acetylation resulted in a more or less uniform distribution of acetyl groups along the anhydroglucose chain.

Four samples of acetates prepared in this way were subjected to microbiological attack in shaker

TABLE IV. DEGRADATION OF CELLULOSE ACETATES BY MICROORGANISMS

Sample No.	Description	% acetyl	Degree of polymerization	*M. verrucaria*	*A. flavipes*	*G. convoluta*	*C. lunata*	*S. myxococcoides*
1	High acetyl, high D.P.	37.9	510	0.0	0.0	0.0	0.0	0.0
2	High acetyl, low D.P.	40.0	100	0.0	0.0	0.0	0.0	0.0
3	Low acetyl, high D.P.	4.8	310	68.5	41.0	37.6	18.6	48.4
4	Low acetyl, low D.P.	3.2	150	64.9	34.2	39.7	13.4	52.8

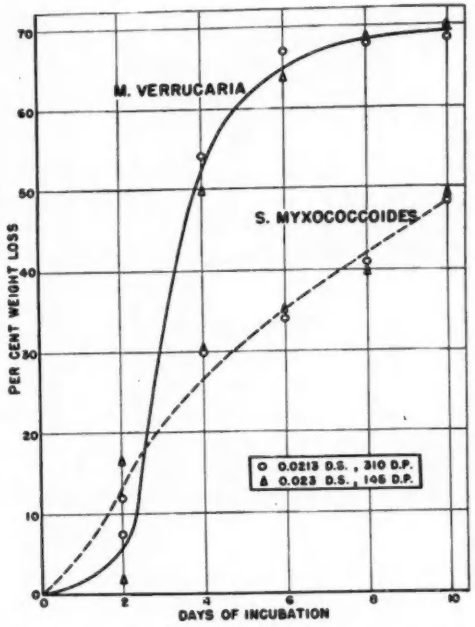

FIG. 1. *Growth of microorganisms on cellulose acetate.*

flasks. The susceptibility of the preparations to these microorganisms during a period of 10 days at 30°C is given in Table IV.

Results of experiments on rate of growth were conducted with *S. myxococcoides* and *M. verrucaria* and are plotted in Figure 1. Similar experiments with triacetates varying in D. P. from 61 to 510 all showed complete resistance to microorganisms.

Discussion

The results given in Table I reveal the high specificity of microorganisms in the utilization of sugar derivatives. Whereas the unsubstituted glucose, mannose, cellobiose, and the glucoside supported growth, any substitution on the pyranose ring resulted in increased resistance to microbiological attack. Apparently neither the nature nor the number of substituents per ring determines the extent of resistance. Any modification of the ring prevents the microorganisms which have been studied from attacking the substrate. The same situation seems to obtain in the case of derivatives of cellulose, as revealed by the data in Table II.

At the same time, however, data in Table II suggest that complete resistance is exhibited only by those preparations with a degree of substitution above one. This indication is further supported by the measurements recorded in Table III. In these experiments it may be seen that samples with 1.43 or more substituents per anhydroglucose unit are all completely resistant to attack by *Myrothecium verrucaria*. Although pure cellulose was digested by the organism to the extent of 80 percent in 13 days, only 29 percent of a cyanoethylcellulose sample (average of 1.02 substituents per anhydroglucose unit) was similarly degraded in the same length of time. Inasmuch as the cyanoethylation was carried out on native fibrous cellulose, it is likely that the sample was not uniform—*i.e.*, it is probable that the loss in weight of 29 percent reflects the behavior of the unreacted fraction of the cellulose in the sample, or, as will be pointed out in the subsequent paragraph, the unsubstituted anhydroglucose units.

Further insight into the mechanism of breakdown of the cellulose molecule may be inferred by examination of the data given in Table IV and Figure 1. The degree of substitution in samples Nos. 3 and 4 represents approximately one substituent of every four anhydroglucose units. Because of the method used, which involves homogeneous acetylation of cellulose in solution, it is reasonable to assume that these substitutions are uniformly distributed along the chain. Total utilization of all the unsubstituted anhydroglucose units in these derivatives should yield a value of approximately 74 percent of their loss in weight. As shown in Figure 1, the actual experimental value approximates 70 percent. The rather close agreement strongly suggests that the organism can attack a glycosidic linkage or an unsubstituted anhydroglucose unit at any position in the cellulose linear chain. It need not *initiate* the attack at the ends, and our interpretation is contrary to Clayson's belief [1] that the cellulolytic action on the molecule proceeds end-wise.

An interesting speculation can be made as to whether the end or the internal linkages are the more susceptible to degradation. If it is assumed that acetylation occurs at random throughout the anhydroglucose units in the cellulose chains (samples Nos. 3 and 4), it then follows that with the same degree of acetylation a sample with a smaller chain length will have a greater proportion of un-

substituted anhydroglucose units at the ends of the chains. The fact that equal rates of utilization of the two samples occur seems to imply that free anhydroglucose units are equally susceptible to attack by microorganisms regardless of their position in the substituted chain. Unfortunately, the differences in chain length between the two substrata are too small and the degree of acetylation is too high to provide a critical evaluation of this hypothesis.

The importance of these theoretical considerations on the potentiality of "multipurpose" topochemical reactions on cotton fibers is highly significant. It appears that in order to impart mildew-resistance to cotton fibers it is necessary only that all anhydroglucose units of the cellulose molecules in the *surface fiber* layers be substituted. Furthermore, it is evident that only one substituent per anhydroglucose unit is necessary. Fortunately, as has been shown, the nature of the substituents may be varied within wide limits. Considerable promise exists, therefore, that from the wide array of possible reactants a number may be selected on the basis of ability to impart additional desirable properties to the topochemically modified cellulosic fabric.

Summary

Experiments on the growth of various selected species of fungi and bacteria isolated from deteriorated cotton textiles uniformly reveal their inability to utilize derivatives of glucose, mannose, cellobiose, and cellulose. Despite a wide range of substituents on various carbohydrates, almost complete, if not complete, resistance to microbiological attack was exhibited by the compounds tested.

In the case of cellulose, the available data strongly suggest that microorganisms are capable of digesting unsubstituted anhydroglucose units at any position in the chain of a partially substituted cellulose derivative.

Literature Cited

1. Clayson, D. H. F., *J. Soc. Chem. Ind. (London)* **62**, 49 (1943).
2. Dorée, C., *Biochem. J.* **14**, 709 (1920).
3. Goldthwait, C. F., McLaren, J., and Voorhies, S. T., Jr., *Textile World* **96**, 115 (Feb. 1946).
4. Heuser, E., Shockley, W., Adams, A., and Grunwald, E. A., *Ind. Eng. Chem.* **40**, 1500 (1948).
5. Kluyver, A. J., and Perequin, L. H. C., *Biochem. Zeit.* **266**, 68 (1933).
6. Reese, E. T., *J. Bact.* **53**, 389 (1947).
7. Siu, R. G. H., *Am. Dyestuff Reptr.* **36**, P320 (1947).
8. Thaysen, A. C., *Brit. Pat. 399,952* (Oct. 19, 1933); *489,901* (June 13, 1938).
9. Thaysen, A. C., Bunker, H. J., Butlin, K. H., and Williams, L. H., *Ann. Applied Biol.* **26**, 750 (1939).

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Detergency Studies*

IV: Influence of Oily Soil upon the Removal of Pigment Soil

William P. Utermohlen, Jr., Earl K. Fischer, Mary E. Ryan, and Gordon H. Campbell

Institute of Textile Technology, Charlottesville, Virginia

Abstract

Natural soils on textiles usually contain both oily and solid components, and the generally accepted mechanism of soil removal postulates the emulsification and suspension of oil droplets or of solid particles covered with an intact oil film. Thus the ready removal of solid soil has been considered as dependent upon the presence of an emulsifiable oily soil, which had also served, prior to washing, to bind the solid soil more tightly to the fabric.

Washing tests have been made on samples of cotton cloth soiled to the same extent with lampblack or iron oxide in the presence of an oily liquid binder, of non-oily, water-soluble liquid binders, and of no binder at all. The ease of removal of the pigments was equal or only slightly different in all of these cases, indicating that the removal of solid soil and of oily soil are separate phenomena and not interrelated to any great extent. This result indicates the desirability of reconsidering the theory of soil removal, and a possible improvement and simplification of laboratory test methods for detergent action.

Introduction

The soil found on textile materials contains several components: water-soluble materials, which offer no real cleaning problem, since they can be easily rinsed away; insoluble solid substances such as lint, dust, and soot; and insoluble oily matter. These two types of insoluble soils necessitate a cleaning treatment which will cause wetting, loosening, and removal of the soiling materials into the washing medium. The oily portion is naturally adherent and causes otherwise loosely adherent solid soil to be bound to the fabric. Because of the frequent coexistence of oily and solid matter in natural soils, it has been generally assumed that synthetic soils should contain these two types of material [1, 4, 6, 7] in order to bring laboratory research work in textile detergency closer to actual practice in laundries and finishing plants.

The effort to simulate natural soils in laboratory testing has influenced theories of the mechanism of removal of mixed oil-solid soils, since it has led to the assumption that the same oil which binds the solid to the fabric plays an important part in the effective removal of the solid soil during washing. It is generally considered that the oil forms a film on the solid or pigmenting components and binds them to the fiber surface. The problems of removal then are those chiefly associated with the water-oil interface, and removal of the composite soil is accomplished by emulsification. This view is either expressed or implied in the work of a number of investigators, of whom Snell [6, 7, 8], Holland and coworkers [1, 2], and Robinson [5] may be mentioned.

It appears, however, that this concept of soil removal is inadequate. No experimental work can be cited in support of the idea that the oil component of natural soil is the predominant factor in removal by detergent processes. Soiling of fabrics occurs with materials containing inappreciable quantities of oily matter. Moreover, the removal of the oily and of the solid components have been shown to be separate processes in the case of wool [3] (Palmer, noting unpublished experiments of Cunliffe), from which oil can be readily removed by washing, while most of the carbonaceous soil is left on the fibers.

The objective of the experiments reported in this paper was to determine the effect of oily components on the removal of pigmenting materials from fabrics.

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Experimental Methods and Materials

Types of Soiling Mixtures

The soiling mixtures used in previous work in this laboratory [9] were composed of a pigment (lampblack or magnetic iron oxide), mineral oil, and hydrogenated cottonseed oil dispersed in carbon tetrachloride, and hence have been similar to the soiling mixtures used by many earlier workers.

In order to compare the removal of oily and non-oily pigment soil, a new series of pigment dispersions in which the oil content varied from that of a "standard" soiling mixture to zero concentration was prepared and used for soiling mixtures. This series was made up with a constant ratio of liquid to solid in the soiling mixtures, the oil in the liquid being progressively replaced by a non-oil diluent. This procedure was considered preferable to diminishing the amount of oil without adding a diluent, as some of the pigment pastes had to be mulled to obtain proper pigment subdivision, and mulling of pastes of widely varying liquid-pigment ratios would not produce the same degree of subdivision. For the diluent to be suitable, the following properties were considered to be necessary: (1) solubility in carbon tetrachloride (the dispersing medium) so that pigment dispersions could be made readily; (2) solubility in water (the washing medium) so that the method of removal of the diluent from the cloth would be by solution rather than by emulsification; (3) compatibility with the mineral oil-hydrogenated cottonseed oil mixture; and (4) a fairly high boiling point and low volatility. "Butyl cellosolve" (ethylene glycol mono-butyl ether) met the necessary requirements, and it was used in a series of soiling mixtures with the oil mixture and pigment. Comparative washings were carried out on swatches of cotton cloth soiled to the same extent (as measured by a photometer) with these various soiling mixtures. When lampblack was used as the soiling pigment, the soil containing the lowest quantity of butyl cellosolve was removed slightly more readily than the soil or soils containing a larger quantity of the butyl cellosolve; the differences, however, were slight. When magnetic iron oxide was used as the pigment, the pieces soiled to the same extent with mixtures of varying oil content were cleaned to the same extent.

Following this work, it was thought desirable to make several changes in the soiling systems, in order to make the results obtained of wider significance. The following variations were introduced:

1. Soiling mixtures were made from a pigment and a liquid binder as described above, but the liquid was either 100 percent oil (water-insoluble) or 100 percent water-soluble material. The materials chosen, in addition to butyl cellosolve (an ether-alcohol) to replace the oil, were 2-ethylhexanediol (a glycol), and "diethyl carbitol" (a polyether, the diethyl ether of diethylene glycol).

2. A dispersing solvent other than carbon tetrachloride and capable of dissolving appreciable amounts of water, the oil liquid, and the non-oil liquids was used. A mixture of equal parts by volume of *n*-butyl alcohol and dioxane was found to be suitable for this purpose.

3. Soiling dispersions were also made containing pigment only (no binder liquid).

In using these soiling mixtures, the application of the soil was adjusted to obtain equal soiling (equal photometer readings) of the various pieces of unwashed cloth, so that their apparent soil contents after washing could be compared more accurately. The pigments which had been dispersed with the water-soluble binders, or with no binding liquid at all, were removed as well, or almost as well, as those dispersed with the oil mixture.

Soiling with a pigment in an aqueous system in the presence or absence of an oil was also tried, using a water dispersion of a carbon black ("Aquablak B"), as described by Vaughn and Smith [11], with or without the addition of a solubilized cutting oil ("Dromus Oil B"). In this case, as in the work previously discussed, pieces soiled to the same apparent extent with the two soiling dispersions were cleaned to the same extent when subjected to a standard wash test.

Soiling and Washing Procedures

The general methods of preparation and application of soiling mixtures, storage and washing of cloth, and estimation of soil content were essentially the same as described earlier [9]. An Atlas Style B-1 Launder-Ometer was used for the washing tests. Several variations in the earlier procedure were tried and found to add to the reproducibility or convenience of soiling and washing tests:

1. An individual wash time of 10 minutes was found to be virtually as effective as periods of 15 or 20 minutes.

TABLE I. Formulas and Soiling Performance of First Series of Soiling Mixtures (Carbon Tetrachloride Solvent)

	Formula No.	Nujol	Snowdrift (parts by weight)	Butyl cellosolve	Reflectivity values (C_f) of soiled pieces of cloth (range)	(average)
Lampblack*	1	10.0	10.0	0.0	17–23	19.5
	2	5.0	5.0	10.0	21–28	24.5
	3	2.5	2.5	15.0	22–27	25.3
	4	1.25	1.25	17.5	25–30	27.8
	5	0.625	0.625	18.75	26–31	28.4
	6	0.0	0.0	20.0	28–33	30.3
Iron Oxide†	7	8.0	8.0	0.0	31–37	33.5
	8	4.0	4.0	8.0	32–40	36.7
	9	2.0	2.0	12.0	36–44	41.1
	10	1.0	1.0	14.0	38–45	42.1
	11	0.5	0.5	15.0	40–48	44.0
	12	0.0	0.0	16.0	42–51	46.2

* Formulas 1–6—Germantown lampblack, 2.22 parts by weight.
† Formulas 7–12—Mapico Black iron oxide, 4.00 parts by weight.

2. The 3-inch swatches from each strip of soiled cloth were sorted according to their individual percentage reflectance values, since it was found that areas in carefully soiled strips of cloth might vary 5 or 6 units in percent reflectance, thus causing a possible error of 2 or 3 percentage units if the average reflectance value for the entire strip was arbitrarily assigned to all the pieces from it. Pieces from a given strip possessing different reflectances were subjected to identical washings, and it was observed that part of the difference in initial reflectance values was carried over to the reflectance values obtained on the test pieces after washing. This indicated that reliable comparisons of reflectance values of washed pieces was possible only by operating on cloth samples soiled to the same reflectance level, as well as from the same soil dispersion. Accordingly, each group of comparative wash tests reported in this paper was carried out on pieces of cloth whose individual or average (of 4 pieces in a particular wash jar) initial reflectance values were equal.

3. Because of the mechanics of agitation of 3-inch square swatches of cloth, $\frac{1}{4}$-inch steel balls, and detergent dispersion in pint Launder-Ometer jars, the swatches often showed crease lines through their centers, and the center areas were washed slightly cleaner than were the corners. Center readings were never used, therefore; four corner readings were made on each washed swatch.

Materials

Aquablak B, Germantown lampblack No. 12, and Mapico Black pigments were obtained from the Binney and Smith Co. Aquablak B is an aqueous dispersion of a fine channel (carbon) black containing 35 percent of the pigment by weight. Mapico Black is a black ferro-ferric oxide containing 99 percent of the pigment by weight. Dromus Oil B was obtained from the Shell Oil Co. It is a water-emulsifiable cutting oil. Butyl cellosolve, 2-ethylhexanediol, diethyl carbitol, *n*-butyl alcohol, and dioxane were obtained from the Carbide and Carbon Chemicals Corp. The detergents employed in the washing tests were used as supplied by the manufacturers. The cloth was a bleached, kiered, desized 80 x 80 nainsook cotton.

Photometric Measurements: Analyses

Reflectance values were measured by means of a Model 610 Reflection Meter made by the Photovolt Corp. The instrument was calibrated against standard reflectance panels obtained from the National Bureau of Standards. Reflectances are expressed relative to that of magnesium oxide, taken as 100 percent.

Iron oxide on the cloths soiled with Mapico Black was determined by ashing and colorimetric analysis, using the orthophenanthroline method. The intensity of the colored solution was compared with standards containing known amounts of the same pigment.

Experimental Results and Discussion

The first sets of soiling mixtures used had the compositions given in Table I. Five g. of each lampblack mixture (or 5.5 g. of each iron oxide mixture) were dispersed in 500 ml. of carbon tetra-

TABLE II. EXTENT OF SOILING AND IRON OXIDE CONTENT OF SOILED CLOTH (INITIAL CONCENTRATION OF BLACK IRON OXIDE EQUAL IN ALL SOILING MIXTURES)

Formula No.	Reflectance (%)	Relative iron oxide content of soiled cloth (mg./2 g. cloth)
7	32	9.7
7	33	9.5
8	35	9.4
8	38	7.9
8	39	8.7
9	39.5	8.2
9	41	7.0
10	42.5	6.4
11	47	5.1
12	45.5	4.6
12	48.5	4.4

chloride; this quantity of dispersion served to soil one 6 x 144-inch strip of cotton cloth. The soiled strips were cut into 3-inch squares, and the percent reflectivity values were measured for the squares. The range and the average of the reflectivity values are shown in Table I.

With both the lampblack and the iron oxide pigments, the apparent extent of soiling decreased as the butyl cellosolve content of the soiling dispersion increased, although all the soiling dispersions contained the same amount of pigment. It was thought that this might be attributed to better dispersion (greater deflocculation) of the pigment in the carbon tetrachloride dispersions containing butyl cellosolve, and thus to lessened concentration of pigment on the surface of the cloth. This was confirmed for the iron oxide soilings by ashing the soiled but unwashed samples of varying reflectance values and determining the relative iron oxide content of the ash. These values, listed in Table II, showed that the iron oxide content decreased as the reflectance increased. This result suggests that the pigment was progressively more deflocculated as the butyl cellosolve content was increased, and that it was deposited in progressively smaller amounts on cloth strips passed through the dispersions.

The soiled samples were washed with a 0.25-percent dispersion of Ivory Snow soap to obtain comparative data on the various soiling mixtures. The lampblack-soiled samples were given two 10-minute washings, while those soiled with iron oxide were given one such washing. Tables III and IV show the reflectance values after washing sets of samples of the same initial reflectance, selected from as many different soiling formulas as possible. The ease of removal of lampblack soil decreased slightly as the butyl cellosolve content of the soiling dispersion increased. The ease of removal of the iron oxide soil did not appear to vary with changes in the butyl cellosolve content of the dispersions.

TABLE III. REFLECTANCE OF PIECES OF LAMPBLACK-SOILED CLOTH AFTER WASHING (0.25% IVORY SNOW)

Formula No.	Initial reflectance* (%)	Reflectance after washing (%)
1	21	61.6
2	21	60.3
1	22	61.2
2	22	61.0
3	22	59.5
1	23	62.9
2	23	61.0
3	23	63.3
2	24	62.0
3	24	61.2
2	25	63.4
3	25	62.7
4	25	61.3
2	26	64.1
3	26	63.9
4	26	62.4
5	26	62.6
2	27	63.8
3	27	64.6
4	27	62.0
5	27	62.1
2	28	64.9
4	28	63.6
5	28	62.2
6	28	62.1
4	29	63.3
5	29	63.0
6	29	62.4
4	30	63.3
5	30	62.8
6	30	62.7
5	31	63.4
6	31	62.5

* Samples of soiled cloth having the same reflectance were selected for comparison within each group.

TABLE IV. REFLECTANCE OF PIECES OF IRON OXIDE-SOILED CLOTH AFTER WASHING (0.25% IVORY SNOW)

Formula No.	Initial reflectance* (%)	Reflectance after washing (%)
7	35	64.4
8	35	61.1
7	36	65.7
8	36	62.0
9	36	62.0
8	37	62.8
9	37	63.1
8	38	63.3
9	38	62.3
8	39	63.5
9	39	63.0
10	39	65.7
8	40	65.0
9	40	66.6
10	40	64.3
11	40	65.5
9	41	64.8
10	41	66.2
11	41	66.7
9	42	65.4
10	42	65.6
11	42	65.7
12	42	65.2
9	43	65.7
10	43	64.2
11	43	64.9
12	43	63.7
9	44	65.9
10	44	65.1
11	44	67.0
12	44	64.2
10	45	67.8
11	45	67.7
12	45	66.3
11	46	67.6
12	46	67.8
11	47	69.8
12	47	68.3
11	48	68.6
12	48	68.1

* Samples of soiled cloth having the same reflectance were selected for comparison within each group.

This work was repeated with a smaller number of soiled swatches, using a 0.25-percent dispersion of Duponol ME (a sodium alkyl sulfate) in place

TABLE V. REFLECTANCE OF PIECES OF LAMPBLACK-SOILED CLOTH AFTER WASHING (0.25% DUPONOL ME)

Formula No.	Initial reflectance* (%)	Reflectance after washing (%)
1	23	56.0
2	23	54.5
3	23	55.9
2	24	55.7
3	24	56.4
2	25	56.3
3	25	57.5
2	26	58.1
3	26	56.7
4	26	55.5
4	27	55.1
5	27	54.7
4	28	56.8
5	28	56.6
6	28	56.6
4	29	57.9
5	29	57.2
6	29	57.4
4	30	58.0
5	30	57.4
6	30	56.1
5	31	56.7
6	31	56.5

* Samples of soiled cloth having the same reflectance were selected for comparison within each group.

TABLE VI. REFLECTANCE OF PIECES OF IRON OXIDE-SOILED CLOTH AFTER WASHING (0.25% DUPONOL ME)

Formula No.	Initial reflectance* (%)	Reflectance after washing (%)
9	41	59.4
10	41	59.6
9	42	61.3
10	42	61.6
9	43	61.0
10	43	60.7
10	44	62.8
11	44	63.2
10	45	61.6
11	45	60.6

* Samples of soiled cloth having the same reflectance were selected for comparison within each group.

of Ivory Snow. With this detergent, the ease of soil removal of both soiling pigments was the same for the various soiling dispersions represented (Tables V and VI).

Because the differences observed in washing behavior of successive soiling formulas were slight, it was decided that further soiling mixtures would be made with either 100 percent oil or 100 percent non-oil as the binder (*i.e.*, mixed compositions would be eliminated). Experiments with such mixtures showed that it was possible to match the initial reflectance values of strips soiled with either the pigment-oil or the pigment-butyl cellosolve mixtures by adjusting the concentration of soiling paste in the carbon tetrachloride dispersant. Strips of cloth were soiled to the same extent (as measured by the photometer) with mixtures containing lampblack as the pigment and either Nujol-Snowdrift mixture or butyl cellosolve as the binding liquid (Formulas 13 and 14), and given two 10-minute washings in 0.25-percent dispersions of Ivory Snow, Duponol ME, and Nacconol NR (a sodium alkyl-arylsulfonate). The reflectance values after these washings are given in Table VII; these results indicated little or no difference in the ease of removal of the two types of soil.

The experiments described indicated that extreme variations in formulation of soiling mixtures resulted in approximately the same degree of removal of pigment soil by washing. This apparent contradiction of accepted theories of soil removal called for further tests in which new soiling formulas were used. These contained a different dispersing medium and new non-liquid components. It was desired that the dispersing liquid should be capable of dissolving appreciable amounts of water, oil, and non-oil binders; it should be inert and should evaporate readily. Methyl, ethyl, and isopropyl alcohols were unsatisfactory for this purpose, but a mixture of equal parts of butanol and dioxane by volume met all the requisites. The compatibility and other requirements of the non-oil binders were the same as those given earlier in the explanation of the use of butyl cellosolve, and were met by 2-ethylhexanediol and "diethyl carbitol" (diethylene glycol diethyl ether). The final soiling mixture of this group contained no binder liquid at all. The soiling mixtures in this series were made with lampblack only as the pigment, because satisfactorily deflocculated stable suspensions of the iron oxide in butanol-dioxane could not be prepared.

TABLE VII. COMPARATIVE RETENTION OF LAMPBLACK SOIL APPLIED WITH DIFFERENT BINDERS (AFTER WASHING IN 0.25% DETERGENT DISPERSIONS)

Formula No.	Binder	Detergent	Reflectance after washing* (%)
13†	Nujol-Snowdrift	Ivory Snow	58.6
14‡	Butyl cellosolve		57.6
13	Nujol-Snowdrift	Duponol ME	56.4
14	Butyl cellosolve		53.2
13	Nujol-Snowdrift	Nacconol NR	51.6
14	Butyl cellosolve		51.4

* Initial reflectance = 32.5% for all pieces.

† Formula 13—Four g. of a mixture containing 45 parts Nujol, 45 parts Snowdrift, and 10.0 parts lampblack was used for 500 ml. carbon tetrachloride (dispersing solvent).

‡ Formula 14—Fifteen g. of a mixture containing 40 parts butyl cellosolve and 4.44 parts lampblack was used per 500 ml. carbon tetrachloride.

The reflectance value obtained after triple passage of a cloth strip through the usual concentration of lampblack and mixed oils in butanol-dioxane was taken as the standard, and other strips were soiled to approximately the same reflectance with the soiling mixtures containing 2-ethylhexanediol, diethyl carbitol, or no binding liquid, by suitable adjustment of the concentrations of the soil dispersions. When this was done, a range of reflectance values was obtained among the pieces from a given soiled strip, but the ranges overlapped well enough so that a sufficient number of pieces of a given average reflectance from each of the soiled strips could be selected for washing (Table VIII).

Pieces of the strips soiled with the mixtures shown in Table VIII were given two 10-minute washings in the same detergent dispersions as used previously. The reflectance values of the cloth pieces so washed are given in Table IX. Only a small lowering in the ease of pigment soil removal was produced by the substitution of a water-soluble binding liquid for the customary oil mixture, or by the omission of the oil or other binding liquid when Ivory Snow or Duponol ME was the detergent; no lowering was noted in the case of Nacconol NR.

The ease of removal of a pigment applied from an *aqueous* dispersing system, in the presence and in the absence of an oil, was then measured. A reference dispersion of Aquablak B carbon black

TABLE VIII. FORMULAS AND SOILING PERFORMANCE OF SECOND SERIES OF SOILING MIXTURES (BUTANOL-DIOXANE SOLVENT, LAMPBLACK PIGMENT)

Formula No.	Soiling formula (parts by weight)	Soiling mixture used (g./500 ml. solvent)	Pigment used (g./500 ml. solvent)	Reflectivity values (%) of soiled pieces of cloth (range)	(average)
15	45 Nujol 45 Snowdrift 10 pigment	20.0	2.0	32–38	34.8
16	40 ethylhexanediol 4.44 pigment	20.0	2.0	32–38	34.2
17	45 diethyl carbitol 10 pigment	13.0	2.4	31–36	33.4
18	Pigment alone	2.5	2.5	33–40	36.0

was prepared by diluting 28.6 g. of the commercial paste to 1 liter with distilled water. Two and one-half ml. of this dispersion and fifteen ¼-in. steel balls were added to 102.5 ml. of water in each of a group of Launder-Ometer jars, and the jars were rotated for 5 minutes to ensure uniform pigment distribution (Formula 19). Two 3-inch squares of clean cloth were then added to each jar and the whole was rotated for 15 additional minutes at 122°F. The samples were then rinsed, dried, and ironed, and measurements were made of their reflectance values. A soiling range of 38 to 41 percent reflectance was obtained. For soiling in the presence of oil, the above procedure was repeated, except that 1.5 ml. of the distilled water was replaced with 1.5 ml. of Dromus Oil B (Formula 20). This provided a soiling range similar to that obtained from Formula 19. Washing of the pieces soiled from these aqueous dispersions was done with the same detergents used before, and again samples of matching initial reflectance were chosen from the two soiled batches. The results of these washings are shown in Table X. Again, no appreciable enhancement of soil removal resulted from the addition of oil to the soiling mixture.

TABLE IX. COMPARATIVE RETENTION OF LAMPBLACK SOIL APPLIED WITH DIFFERENT BINDERS (AFTER WASHING IN 0.25% DETERGENT DISPERSIONS)

Formula No.	Binding liquid	Detergent	Reflectance after washing* (%)
15	Nujol-Snowdrift	Ivory Snow	64.6
16	2-Ethylhexanediol		64.3
17	Diethyl carbitol		60.4
18	None		60.2
15	Nujol-Snowdrift	Duponol ME	60.4
16	2-Ethylhexanediol		58.7
17	Diethyl carbitol		56.6
18	None		56.4
15	Nujol-Snowdrift	Nacconol NR	55.5
16	2-Ethylhexanediol		57.1
17	Diethyl carbitol		54.3
18	None		56.3

* Initial reflectance = 34.5 ± 0.3% for all pieces.

TABLE X. COMPARATIVE RETENTION OF A CARBON PIGMENT APPLIED FROM AQUEOUS DISPERSION WITH AND WITHOUT AN OIL BINDER (AFTER WASHING IN 0.25% DETERGENT DISPERSION)

Soiling Formula No.	Soiling system	Detergent	Initial reflectance (%)	Reflectance after washing (%)
19	Aquablak B alone	Ivory Snow	38.5	46.9
20	Aquablak B plus Dromus Oil B		38.5	44.3
19	Aquablak B alone	Duponol ME	39.5	46.6
20	Aquablak B plus Dromus Oil B		39.5	45.3
19	Aquablak B alone	Nacconol NR	40.0	44.8
20	Aquablak B plus Dromus Oil B		40.0	46.4

The results of detergency experiments described above indicate that the presence of oils in the artificial soiling mixtures caused little or no increase in the ease with which the pigment soil could be removed, despite general statements to the contrary in the literature. Although pigment particles may be aided by an oily vehicle in their attachment to fabrics at the time of soiling, emulsification of this binding oil is not a sufficient explanation for the action of detergents on soiled fabrics. The reflectance data in Tables VII, IX, and X show that drastic changes in the formulation of the applied soil—use of an oil binder, of a non-oil binder, or of no binder liquid at all—resulted in little or no difference in the amount of pigment soil removed by the detergent processes used. It is especially significant that soil composed of pigment alone was removed about as readily as were soils composed of pigment plus either oil or non-oil binders. Different types of detergents exhibited comparable effects in this respect. The conclusions seem justified that the removal of oil soil and of pigment soil from cotton cloth are largely independent processes for artificial soils, and that the pigment does not depend for removal upon the presence of an oil. The authors consider it possible that the importance of an oil in aiding the removal of natural soils from cotton fabrics is also slight or negligible, and believe that the previously postulated theories of the mechanism of removal of solid soil from textile materials are in need of reconsideration.

If the presence of an oil component in artificial soiling mixtures has no value in aiding in the removal of the solid pigment soil by washing, it might as well be omitted from the soiling dispersion. This omission would automatically eliminate the changes in the ease of cleaning of artificially soiled fabrics upon ageing, which are due to polymerization of the unsaturated components of the oil [10]. Thus, a simplification of soiling formulations and an improvement in the uniformity of the test material could be accomplished at the same time.

Summary

1. A series of artificial soiling mixtures was applied to cotton cloth to determine the effect of oil components in the mixtures on the retention of the pigment component when the cloth was subjected to laboratory washing procedure. These mixtures were of the following types: (*a*) conventional-type formulations in an organic liquid dispersion, in which the oily components were progressively replaced by non-oily liquids, maintaining a constant ratio of pigment to liquid; (*b*) similar formulations in which the entire "binding" liquid was a water-soluble material; (*c*) similar formulations in which no binder liquid was used; (*d*) aqueous dispersions of carbon black, both with and without the addition of a solubilized oil.

2. Pieces of the cloths soiled with the above mixtures were washed with three different types of detergents. The apparent pigment soil removal was remarkably similar for the various groups of comparable soils, when pieces of equal initial soil content were compared. Pigment soil applied with an oily binder, a non-oily binder, or no liquid binder was removed to the same extent.

3. It is concluded that the removal of oily soil and of pigment soil from cotton cloth are largely independent processes for artificial soils, and possibly for natural soils, and that the pigment does not depend for removal upon the presence of an oil. This indicates a necessity for reconsidering the theories concerning the removal of solid soil from textile materials.

Literature Cited

1. Clark, J. R., and Holland, V. B., *Am. Dyestuff Reptr.* **36**, 734–47 (Dec. 15, 1947).
2. Holland, V. B., and Petrea, A., *Am. Dyestuff Reptr.* **32**, 534–7 (Nov. 22, 1943).
3. Palmer, R. C., *J. Soc. Chem. Ind.* **60**, 56–62 (Mar. 1941).
4. Rhodes, F. H., and Brainard, S. W., *Ind. Eng. Chem.* **21**, 60–8 (1929).
5. Robinson, C., Chapter in "Wetting and Detergency," pp. 137–51 (esp. p. 137), London, A. Harvey, 1937.
6. Snell, C. T., *J. Chem. Ed.* **24**, 505–11 (1947).
7. Snell, F. D., *Food Industries* **13**, No. 10, 48–50 (1941).
8. Snell, F. D., *Ind. Eng. Chem.* **35**, 107–17 (1943).
9. Utermohlen, W. P., Jr., and Wallace, E. L., TEXTILE RESEARCH JOURNAL **17**, 670–81 (1947).
10. Utermohlen, W. P., Jr., and Wallace, E. L., TEXTILE RESEARCH JOURNAL **17**, 682–88 (1947).
11. Vaughan, T. H., and Smith, C. E., *J. Am. Oil Chem. Soc.* **25**, No. 2, 44–51 (Feb. 1948).

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INDUSTRIAL SECTION

Wool Damage Data

Kermit S. LaFleur

Farnsworth Mill, Lisbon Center, Maine

Abstract

Rough categories into which wool damage may be classified for study include hydrolysis, reduction and oxidation effects, and incipient decomposition caused by exposure to light or high temperatures in the presence of air. Data have been accumulated which describe some specific effects obtained by treating woolen swatches in boiling water as a function of time, in boiling acid (sulfuric) or alkaline (sodium hydroxide) solutions over a wide pH range, in boiling reducing (thioglycollic acid, zinc formaldehyde sulfoxylate) or oxidizing (hydrogen peroxide) solutions over a wide rH range, and upon drying at 260°F for periods ranging to 128 hours. Quantitative measures of wool damage which have been used include loss of weight, area shrinkage, alkali solubility, wettability in water and 0.1*N* sodium hydroxide, and loss of tensile strength.

WOOL is seldom satisfactorily uniform or uniformly susceptible to change [8]. As a biological product, it is, in general, subject to variations which exceed the narrowing tolerances established by increasingly critical quality standards. It follows that whatever the degree of success may be in attempts to preserve existing wool qualities, highly desirable as such preservation is, such attempts cannot be expected to produce a permanently adequate solution to the problem. Improvement of uniformity, equalization of susceptibility to change, as well as good preservation of these gains throughout processing, are objectives which very gradually are becoming mandatory. These and similar needs have already led to the development of modified wool [4, 6, 12] of improved stability, and appear to be leading to the development of regenerated keratin [11, 17, 18] of improved uniformity. Possibly other means will be found to produce a more homogeneous and stable wool [5].

"Damaged" wool is a term descriptive of altered properties, actual and potential, caused by "extraneous" factors not common to all wool being compared, by common factors differing in degree, or by common factors acting upon a non-uniform base. From both theory and observation it seems clear that there can be no "undamaged" wool, and that it is unlikely that there can be uniformly "damaged" wool. "Damage" is a relative term which can have meaning for us only in a statistical sense.*

Methods for estimating wool damage seem prerequisite both for finding methods for controlling it and for attempting to produce improved wools. The purpose of this study is to measure and to compare the changes in woolen swatches caused by hydrolyzing, oxidizing, reducing, and high-temperature treatments † with simple methods which show good differentiation. Modifying treatments designed to improve wool may then be evaluated and interpreted with some assurance; or effective methods for solubilizing keratin with minimum degradation, preparatory to regeneration, may be chosen.

One method of comparison of change, the alkali solubility test [7], originally intended to be specific, more lately has been used rather indiscriminately,

* The implied distinction between non-uniformity in wool caused by "extraneous" factors ("damage") and that caused by biological (growth) factors may be artificial when applied to growing wool, but it is quite valid when applied to the study of wool ready for processing or wool being processed.

† These treatments seem quite representative of wool damage agents.

TABLE I. EFFECTS OF TREATMENT IN BOILING WATER ON WOOLEN SWATCHES

No.	Hrs. boiled	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Loss in weight (%)	Loss in tensile strength (%)	Alkali solubility (%)	Sinking time in water (sec.)	Sinking time in 0.1N NaOH (sec.)	Ml. 0.1N H_2SO_4 absorbed per g. of wool
1	0								12.0	1,226	91	8.9
2	1	6.4	22.6	6.7	23.7	4.6	2.9	20	10.1	60	9	8.6
3	2			6.75	23.75	4.4	3.3	30	9.3	44	7	8.9
4	4			6.8	23.85	3.1	3.7	28	9.3	18	7	9.1
5	8			6.85	24.05	2.2	4.4	34	10.4	22	6	8.9
6	16			6.9	24.2	2.6	5.4	38	12.4	15	5	9.3
7	32			6.9	24.6	0.9	7.9	58	16.5	16	4	10.7
8	64			6.45	24.3	2.0	14	69	34.4	7	4	10.5

probably because it is simple and appears to be significant [14, 15], although by some still obscure mechanism. The comparison of alkali-solubility results with other data contained in this report may help to clarify the applicability of this test.

An earlier paper [9] described the difference in water wettability of woolen swatches exposed to carbon-arc radiation. Later observations indicate that good correlation exists between wettability and incipient damage from other causes, although the method is not satisfactorily precise and does not differentiate degrees of damage beyond initial stages. Wettability in 0.1*N* sodium hydroxide appears to parallel water wettability, but it is considerably faster.

A significant measure of damage in the case of treatments with reducing or oxiding agents is the area shrinkage of the treated swatch. This shrinkage suggests rupture of cystine cross-linkages in the wool fibers, releasing the polypeptide chains to assume less oriented, more probable configurations [11]; the shrinkage is really three-dimensional and cannot be accounted for by the accompanying loss in weight (which suggests a packing effect).

Experimental Procedure

Swatches were cut from a mill-production, unscoured, 12-ounce woolen * fabric previously cleaned in successive baths of petroleum ether at room temperature until the solvent remained clear, followed by successive treatments in water at 100°F until the water remained clear. The swatches were adjusted to an air-dry weight corresponding to an oven-dry (105°C) weight of 2.5 grams, measured for initial area, and entered into 100-to-1 baths of boiling water, or boiling solutions of sulfuric acid or sodium hydroxide, or boiling solutions of thioglycollic acid (to obtain symmetrical reduction of the disulfide linkage), zinc formaldehyde sulfoxylate (to obtain asymmetrical reduction), or hydrogen peroxide. Boiling was carried out under reflux condensers. Additional swatches were entered into thioglycollic acid baths maintained at room temperature or at 150°F, and hydrogen peroxide baths maintained at 150°F.

Initial and equilibrium pH and rH † [2, 3] measurements were obtained for the baths at room temperature with a Beckman pH meter using a glass electrode or a platinum electrode, respectively, with the standard calomel electrode.

The treated swatches were examined for loss of weight, area shrinkage, alkali solubility [7], wettability in water [9], and wettability in 0.1*N* sodium hydroxide by the same method; loss of tensile strength was determined with a Scott motor-driven pendulum-type tester.

The percent acid in swatches treated with hydrogen peroxide was determined by the pyridine method [1] because of the unexpected equilibrium pH values of the solutions.

The combining capacity of wool with acid was determined in the case of swatches boiled in water and acid or alkaline solutions by immersion in 0.1*N* sulfuric acid at room temperature for 16 hours and titration of an aliquot of the equilibrium solution.

Because boiling for even 1 hour greatly modifies the subsequent wettability of wool, shorter immer-

* 30% scoured Cape wool, 20% scoured South American wool, 50% noils.

† rH is an expression of the oxidizing or reducing potential of a solution (in one number which includes a correction for the pH value at which the observation is made).

TABLE II. Effects of Treatment in Boiling Acid or Alkaline Solutions for 2 Hours on Woolen Swatches

No.	Ml. 0.1N H_2SO_4 used per 100 ml.	Ml. 0.1N NaOH used per 100 ml.	Initial pH	Initial rH	Equil. pH	Equil. rH	Loss in weight (%)*	Area shrinkage (%)	Sinking time in water (sec.)	Sinking time in 0.1N NaOH (sec.)	Alkali solubility (%)	Loss in tensile strength (%)	Ml. 0.1N H_2SO_4 absorbed per g. of treated wool
1	90.0		1.25		1.3		5.1	5.6	36	4	27.6	21	10.0
2	71.5		1.35		1.4		4.5	6.0	44	6	23.2	18	
3	53.5		1.45	23.9	1.55	24.0	4.0	5.2	54	3	20.9	16	10.0
4	42.3		1.55		1.65		3.7	5.2	30	3	17.7	14	
5	32.4		1.65		1.75		3.3	4.2	30	4	17.6	12	
6	18.8		1.9		2.05		2.7	6.0	29	4	14.7	9	9.3
7	11.3		2.15		2.4		2.3	5.1	16	5	12.7	5	
8	6.6		2.35	23.9	2.9	24.1	1.6	8.0	16	6	10.0	8	
9	4.0		2.55		3.5		0.9	8.5	12	3	10.4	15	
10	2.35		2.75		4.0		1.1	9.7	18	5	9.7	14	8.8
11	1.55		2.9		4.6		0.9	9.0	24	5	9.6	17	
12	1.2		3.1		4.85		1.0	8.7	30	6	9.6	18	
13	0.55		3.5	22.2	6.1	23.9	1.8	10.3	39	10	8.9	23	
14	0.35		3.85		6.2		1.6	8.9	40	10	9.1	23	
15	0	0	6.6	22.0	6.75	25.0	1.7	8.1	43	10	9.0	30	8.8
16		0.3	10.05		7.0		1.6	8.4	46	8	8.8	26	
17		0.5	10.45		7.1		1.9	10.1	57	10	8.8	26	
18		1.0	10.75		7.4		1.9	11.3	67	11	9.1	31	
19		1.65	10.9	26.1	8.5	25.1	2.4	7.9	28	9	7.9	42	8.3
20		2.85	11.2		9.15		2.9	6.0	43	11	7.8	38	
21		4.1	11.35		9.25		3.7	3.4	39	11	7.2	38	8.5
22		6.35	11.5		9.55		6.6	2.1	33	11	7.7	50	
23		10.3	11.65	26.4	10.5	22.1	13	+3.0	25	11	6.3	54	
24		17.1	11.75		10.7		25	1.7	34	16	7.9	83	10.8
25	Untreated								1,100	90	9.5		8.8

* An excess of 0.1N H_2SO_4 was added to the swatches treated with NaOH, and all swatches were then extracted with pyridine before weighing.

sion periods in boiling water were tried, and swatches so treated were tested for wettability.

Finally, additional swatches were dried at 260°F for varying periods, and tested for wettability in water and 0.1N sodium hydroxide, and for alkali solubility. The percent acid developed during these exposure periods was determined by the pyridine method.

Data are given in Tables I to XII. Each entry is the mean of at least two sets of determinations.

Discussion

Data in Table I show that the solutions tend to become slightly more oxidizing, indicating the possibility that boiling in water has a slight reducing action on wool. Both the alkali-solubility results and maximum-combining-capacity results may indicate incipient hydrolysis upon prolonged boiling (16 hours). Wettability data indicate that the significant factors which affect this test operate within the first hour's boiling, and that the change is not great thereafter.

Table II shows that tensile-strength preservation is greatest at about pH 2.5, but that damage measured by alkali solubility increases inversely as pH, below neutrality. Boiling in acid solution may reduce wool slightly. Minimum loss of weight seems to occur at the isoelectric "point" of wool, which is in better agreement with tensile-strength data than with alkali-solubility data. Wettability does not appear to have much value from these data because the range of damage which it measures with good differentiation has been greatly exceeded. (See also Figure 1.)

Boiling in sodium hydroxide solutions may have a slight oxidizing effect on wool, since solutions are slightly reduced. Alkali solubility is not a measure of alkaline damage.

The increased acid-combining capacity of wool treated in relatively strong acid or alkaline solutions indicates incipient hydrolysis.

Tables III, IV, V, and VI indicate that alkali solubility varies directly with the concentration of thioglycollic acid used and with the duration of

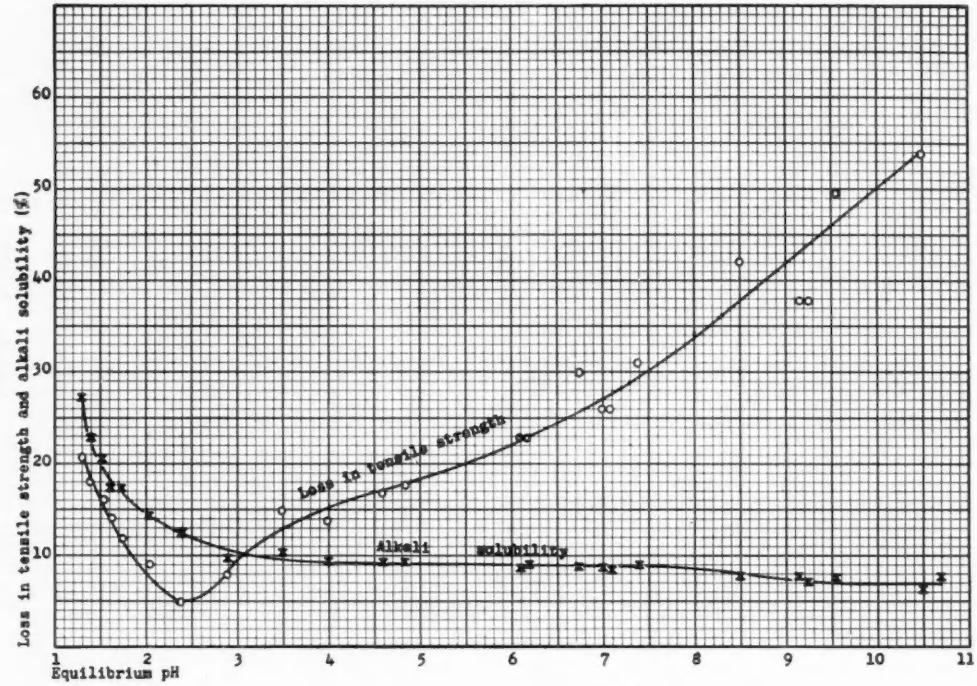

FIG. 1. *Loss in tensile strength and alkali solubility of swatches treated in boiling solutions as a function of pH (see Table II).*

the treatment. Room-temperature experiments are strikingly less effective than treatments at the boil. Neither area shrinkage nor loss of weight appears to be affected significantly by low concentrations of thioglycollic acid, but the effect of higher concentrations is great at the boil or after long treatments at 150°F. In Table VI is found the first example in this report of a range of usefulness of wettability tests under conditions which do not significantly affect the other measures of damage used.

Equilibrium thioglycollic acid solutions in which wool had been treated at the boil became turbid upon cooling, and a white precipitate settled out. Solutions used at room temperature remained clear. A strong odor, suggesting a sulfur derivative, developed in the boiled solutions upon standing. Foam developed in the boiling solutions which was proportional to the thioglycollic acid concentration. This was not true of the boiling hydrogen peroxide solutions.

From Tables III, IX, and X it can be seen that alkali solubility varies directly as the concentration of hydrogen peroxide and with duration and temperature of treatment. Neither loss of weight nor area shrinkage is much affected at low concentrations, but both are strongly increased at higher concentrations. The wettability data of Table X indicate that this test should have some value in differentiating between degrees of mild oxidation, which supports earlier conclusions [9].

The anomalous initial or equilibrium rH values of both thioglycollic acid and hydrogen peroxide solutions can be attributed in part to changes which take place during boiling with no cloth present (Table XIII), but the causes of these changes are not known to the author.

The low pH values of equilibrium hydrogen peroxide baths, compared to initial values, indicate that free acid is being produced, which, unless circumstances are very anomalous, suggests that the

TABLE III. Effects of Treatment in Boiling Thioglycollic Acid or Hydrogen Peroxide Solutions for 2 Hours on Woolen Swatches

No.	Ml. 0.218N thioglycollic acid used per 100 ml.	Ml. 0.1N H_2O_2 used per 100 ml.	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Loss in weight (%)	Sinking time in water (sec.)	Sinking time in 0.1N NaOH (sec.)	Alkali solubility (%)	Loss in tensile strength (%)	Acid as H_2SO_4 in treated swatch (%)
1	80		1.95	8.65	2.15	8.95	41	46	150	5	78	100	
2	40		2.15	9.2	2.35	9.6	33	29	38	2	64	92	
3	20		2.35	10.3	2.55	10.4	31	9.1	29	2	53	63	
4	10		2.5	11.1	2.75	10.8	14	3.8	14	2	29	54	
5	5		2.7	12.15	3.0	12.0	+1.5	1.8	24	3	12	54	
6	0	0	6.8	23.5	6.8	22.9	5.0	2.5	41	7	8.0	33	
7		5	6.8	29.0	4.05	28.2	0	3.6	11	4	47	25	0.47
8		10	6.8	29.1	3.4	29.1	+4.5	7.7	12	7	57	58	0.58
9		20	6.6	29.6	3.15	29.4	51	19	7	2	97	98	1.78
10		40	6.4	29.9	2.9	29.5	61	28	3	2	100	100	3.29
11		80	6.0	31.7	2.9	29.6	72	52	5	2	100	100	3.80

TABLE IV. Effects of Treatment in Boiling Thioglycollic Acid Solutions for 1 Hour on Woolen Swatches

No.	Ml. 0.22N thioglycollic acid used per 100 ml.	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Loss in weight (%)	Sinking time in water (sec.)	Sinking time in 0.1N NaOH (sec.)	Alkali solubility (%)	Loss in tensile strength (%)
1	80	1.95	8.5	2.15	8.2	37	34	43	4	71	87
2	40	2.15	9.15	2.35	8.8	31	13	31	3	64	42
3	20	2.35	9.9	2.55	9.45	18	4.5	16	3	36	29
4	10	2.5	10.55	2.75	10.15	3.0	3.0	26	4	15	33
5	5	2.7	11.3	3.0	10.85	3.3	2.5	40	5	12	35
6	0	6.6	22.9	6.8	22.0	5.3	2.5	53	8	8.0	17

TABLE V. Effects of Duration of Treatment in Thioglycollic Acid Solutions at 150°F on Woolen Swatches

No.	Duration of treatment (hrs.)	Ml 0.248N thioglycollic acid used per 100 ml.	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Loss in weight (%)	Sinking time in water (sec.)	Alkali solubility (%)
1	1	40	2.15	9.05	2.2	9.05	6.5	2.5	—	36
2	4				2.2	9.05	7.5	3.5	49	54
3	9				2.25	9.05	—	—	—	59
4	16				2.3	9.1	34	25	34	72
5	25				2.3	9.1	35	27	20	76

TABLE VI. Effects of Treatment in Thioglycollic Acid Solutions at Room Temperature (77 ± 5°F) for 24 Hours on Woolen Swatches

No.	Ml. 0.207N thioglycollic acid used per 100 ml.	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Loss in weight (%)	Sinking time in water (sec.)	Alkali solubility (%)
1	80	1.95	8.45	2.15	8.0	7.3	1.8	254	21
2	40	2.15	9.05	2.35	8.55	6.4	1.8	797	17
3	20	2.35	9.6	2.6	9.25	5.5	1.8	757	16
4	10	2.5	10.25	2.8	9.8	7.3	1.1	1,347	14
5	5	2.7	11.3	3.1	10.6	7.3	1.1	1,425	14
6	0	6.6	22.2	6.9	23.1	4.5	1.8	1,043	11

woolen swatches have combined with the acid produced to its specific equilibrium value at the given hydrogen ion concentration. Pyridine extraction of the treated swatches supports this conclusion. This reaction may partially explain the decreased combining capacity of wool oxidized with hydrogen peroxide noticed by Harris and collaborators [13, 16].

Table VII indicates that area shrinkage, loss of weight, and alkali solubility are in direct proportion to the concentration of the zinc formaldehyde sulfoxylate used. Initial rH measurements on these solutions do not change significantly upon boiling, which is not true of thioglycollic acid or hydrogen peroxide solutions. Table VIII demonstrates the great dependence of reducing power of zinc formaldehyde sulfoxylate upon hydrogen ion concentration.

Only the data of Tables VII and VIII show the expected migration of rH in reaching equilibrium. Although the usual precautions were taken in making all rH measurements, undesirable variations and unexpected anomalies were experienced. Reference to

TABLE VII. EFFECTS OF TREATMENT IN BOILING ACIDIFIED ZINC FORMALDEHYDE SULFOXYLATE SOLUTIONS FOR 1 HOUR ON WOOLEN SWATCHES

No.	Ml. 0.53N $Zn(HSO_2 \cdot CH_2O)_2$ used per 100 ml.	Initial pH	Adjusted initial pH (H_2SO_4)	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Approx. loss in weight (%)	Alkali solubility (%)	Loss in tensile strength (%)
1	80	3.7	3.0	8.35	2.5	12.5	28	3	78	79
2	40	3.7	2.9	8.85	2.6	15.25	29	3	70	42
3	20	3.7	2.75	9.75	2.8	21.1	28	3	64	
4	10	3.75	2.65	10.85	3.1	21.8	4.3	1	14	
5	5	3.8	2.65	11.95	3.4	22.05	5.1	1	10	
6	2.5	4.6	2.5	15.55	3.6	22.0	5.1	1	10	
7	1.25	5.5	2.5	17.35	3.6	21.45	6.0	1	10	
8	0	6.5	2.5	21.4	3.5	21.7	6.0	1	11	

TABLE VIII. EFFECTS OF TREATMENT IN BOILING ZINC FORMALDEHYDE SULFOXYLATE SOLUTIONS (20 ML. 0.53N PER 100 ML.) FOR 1 HOUR AS A FUNCTION OF pH ON WOOLEN SWATCHES

No.	Ml. 0.1N H_2SO_4 used per 100 ml.	Ml. 0.1N NaOH used per 100 ml.	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Approx. loss in weight (%)	Sinking time in water (sec.)	Alkali solubility (%)
1	35		1.8	13.2	2.0	18.0	4.3	1	447	13
2	20		2.05	13.0	2.45	17.4	+1.7	1	159	11
3	12		2.25	12.8	2.7	16.4	1.7	1	1,198	10
4	4.3		2.75	10.8	3.05	14.7	26	1	11	64
5	1.2		3.05	10.1	3.05	14.4	26	3	33	58
6	0	0	3.05	10.1	3.1	14.3	25	3.5	34	58
7		.25	3.15	10.0	3.1	14.2	25	3.5	45	50
8		1.0	3.3	9.3	3.1	14.0	26	7.5	61	52
9		2.5	4.15	9.3	3.1	13.7	24	7.5	18	47
10		10	7.15	ppt.	3.3	10.5	6.8	2.0	90	55

TABLE IX. EFFECTS OF TREATMENT IN BOILING HYDROGEN PEROXIDE SOLUTIONS FOR 1 HOUR ON WOOLEN SWATCHES

No.	Ml. 1N H_2O_2 used per 100 ml.	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Approx. loss in weight (%)	Sinking time in water (sec.)	Alkali solubility (%)	Acid as H_2SO_4 in treated swatch (%)
1	0	6.8	23.0	6.8	22.5	5.5	4	60	10	
2	5	6.8	29.0	5.3	28.6	4.2	3	18	30	0.25
3	10	6.8	29.1	4.5	29.2	2.0	4	12	57	0.40
4	20	6.6	29.6	3.75	30.0	+1.1	5	7	89	0.75
5	40	6.4	29.9	3.3	30.1	20	6	8	97	1.53
6	80	6.0	31.7	3.0	30.2	56	17	7	100	2.86

TABLE X. Effects of Treatment in Hydrogen Peroxide Solutions (80 ml. 1.N per 100 ml.) at 150°F as a Function of Time on Woolen Swatches

No.	Hrs. at 150°F	Initial pH	Initial rH	Equil. pH	Equil. rH	Area shrinkage (%)	Loss in weight (%)	Sinking time in water (sec.)	Alkali solubility (%)
1	1	6.0	31.5	6.05	31.5	5.2	2.1	—	19
2	4			4.9	31.8	3.0	2.1	395	56
3	9			—	31.7	10	—	18	87
4	16			3.05	31.8	65	45	6	100
5	25			3.1	31.4	69	53	6	100

TABLE XI. Effect of Duration of Immersion Period in Boiling Water on the Wettability of Woolen Swatches

No.	Duration of boiling period (min.)	Sinking time in water (sec.)	Sinking time in 0.1 N NaOH (sec.)
1	0	1,192	86
2	(8 sec.)*	806	48
3	1	600	39
4	2	592	48
5	3	374	24
6	5	147	17
7	10	113	17
8	15	105	17
9	30	81	12
10	45	52	10
11	60	66	10
12	90	53	8
13	120	48	7

* Wet out in boiling water only.

TABLE XII. Effects of Drying at 260°F on Woolen Swatches

No.	Hrs. at 260°F	Acid as H_2SO_4 (%)	Sinking time in water (sec.)	Sinking time in 0.1 N NaOH (sec.)	Alkali solubility (%)
1	0	0	1,270	84	9.6
2	1	0	1,378	217	9.8
3	2	.05	1,638	222	9.0
4	4	.07	2,705	263	10.6
5	8	—	1,752	245	10.4
6	16	.12	1,066	232	10.5
7	32	.22	933	224	11.3
8	64	.29	302	129	11.2
9	128	.44	147	77	13.0

Table XIV (see Table II also) will show that corrections for pH in deriving rH values from E_h (observed potential) measurements are not sufficiently precise, or that the observations themselves are not sufficiently accurate. Drifting of the indicator needle was common, and establishment of equilibrium in each system took several minutes.

Table XI indicates that wettability of wool is increased greatly by relatively short periods of immersion in boiling water. Much of the wettability data gathered so far suggest the possibility of the existence of a relatively impermeable but easily ruptured wool membrane or integument (or the equivalent). A recent paper [10] seems to support such a conclusion, which at this stage is of course purely speculative.

Finally, Table XII shows that initial periods of exposure to high temperatures decrease wettability, whereas continued exposure increases it, all exposures within 128 hours being well within the range where good differentiation is possible.

The validity of the choice of significant figures in

TABLE XIII. Observed Effect of pH Upon the rH Values of Zinc Formaldehyde Sulfoxylate Solutions

No.	Ml. 0.53 N $Zn(HSO_2 \cdot CH_2O)_2$ used per 100 ml.	Initial pH	pH after adding H_2SO_4	Initial rH	rH after adding H_2SO_4	Equil. pH	Equil. rH	Equil. rH after neutralizing
1	80	3.7	3.0	8.7	8.0	2.5	12.8	12.2
2	40	3.7	2.9	9.15	8.5	2.6	15.0	15.5
3	20	3.7	2.75	10.3	9.2	2.8	19.6	22.6
4	10	3.75	2.65	11.4	10.3	3.1	20.6	23.0
5	5	3.8	2.55	12.4	11.5	3.4	20.5	23.6
6	2.5	4.6	2.5	15.2	15.9	3.6	20.8	23.2
7	1.25	5.5	2.5	16.7	18.0	3.6	20.0	22.9
8	0	6.5	2.5	21.4	21.3	3.5	21.0	22.4

TABLE XIV. CHANGES IN rH VALUES OF THIOGLYCOLLIC ACID AND HYDROGEN PEROXIDE SOLUTIONS UPON BOILING WITH NO WOOL PRESENT

No.	Ml. 0.218N $(HS)CH_2COOH$ used per 100 ml.	Ml. 1N H_2O_2 used per 100 ml.	rH before boiling	rH after boiling 1 hr.	rH after boiling 2 hrs.
1	80		8.65	8.1	7.2
2	40		9.2	8.7	8.45
3	20		10.3	9.45	9.35
4	10		11.1	10.05	10.0
5	5		12.15	10.8	10.9
6	0	0	22.9	22.9	22.9
7		5	28.2	30.85	30.8
8		10	29.0	29.35	30.8
9		20	28.9	27.65	30.1
10		40	28.85	28.1	29.6
11		80	28.9	28.8	29.1

expressing such parameters as area shrinkage and loss in tensile strength may be questioned in view of the small size of the swatch and other factors; quite probably these values should be used as guides indicating trends, rather than regarded as absolute. The disturbingly high variability of many comparable measurements on wool indicates how tentative such results are and stresses the need for interpolating broad tolerances until the variables are recognized and corrected for.

Conclusions

A general conclusion from all these data is that the water wettability of wool may be useful for estimating degrees of damage to which many other tests are not sensitive. It also seems that any woolen fabric which has been immersed in boiling water solutions usually should be readily detectable by this method—that is, from its greatly increased wettability. There seems to be little point in using the accelerated wetting of 0.1N sodium hydroxide with lightweight fabrics.

The area shrinkage of woolen swatches treated in reducing or oxidizing solutions seems to be a very instructive test in following the progress of cystine-linkage rupture. It has proven to be useful and reliable in keratin-solubilizing experiments. Probably the method could be developed to have real quantitative significance.

The splitting off of acid in the reaction of wool with hydrogen peroxide and the partial recombination of the wool with the acid is an interesting example of the unexpected results obtained with familiar reactants when used on a different scale.

Literature Cited

1. Barritt, J., *J. Text. Inst.* **26**, T87–92 (1935).
2. Beckman Bulletin 99-B, National Technical Laboratories, South Pasadena, Calif.
3. Coleman Electric Company Bulletin T–117, Maywood, Ill.
4. Geiger, W., Kobayashi, F., and Harris, M., *Am. Dyestuff Reptr.* **32**, 5 (1943).
5. Harris, M., and Brown, A., TEXTILE RESEARCH JOURNAL **17**, 16 (1947).
6. Harris, M., and Brown, A., *Am. Dyestuff Reptr.* **36**, 12 (1947).
7. Harris, M., and Smith, A., *Am. Dyestuff Reptr.* **25**, 20 (1936).
8. Kienle, R., Royer, G., and McCleary, H., Calco Tech. Bull. No. 762 (1944).
9. LaFleur, K., TEXTILE RESEARCH JOURNAL **18**, 1 (1948).
10. Lindberg, J., TEXTILE RESEARCH JOURNAL **19**, 1 (1949).
11. Lundgren, H., TEXTILE RESEARCH JOURNAL **15**, 10 (1945).
12. Patterson, W., Geiger, W., Mizell, L., and Harris, M., *Am. Dyestuff Reptr.* **30**, 17 (1941).
13. Rutherford, H., and Harris, M., *Am. Dyestuff Reptr.* **27**, 7 (1938).
14. Smith, A., and Harris, M., *Am. Dyestuff Reptr.* **25**, 7 (1936).
15. Smith, A., and Harris, M., *Am. Dyestuff Reptr.* **25**, 14 (1936).
16. Smith, A., and Harris, M., *Am. Dyestuff Reptr.* **26**, 15 (1937).
17. U. S. Patent 2,434,688 (R. Evans), Jan. 20, 1948.
18. U. S. Patent 2,436,156 (R. Upson to E. I. du Pont de Nemours), Feb. 17, 1948.

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Correlation of Simulated Rainfall Tests with Laboratory Penetration Tests*

M. I. Landsberg, Roger Kelly, and Dorothy Sinski

Office of The Quartermaster General, Military Planning Division, Research and Development Branch

In an effort to establish a relationship between the length of time a garment will keep the wearer dry and laboratory tests of the fabric from which that garment was made, the study described in this report was undertaken. Since the rainroom has been recognized as a valid testing device, a correlation between rainroom results and data obtained by the much simpler and more economical laboratory tests should in most cases furnish an accurate method of predicting the performance of a garment under conditions of actual wear. This study shows that it is possible to estimate from the results of laboratory penetration tests the most probable rainroom protection time of a fabric treated with a durable water-repellent compound.

Introduction

The need for a convenient and reliable method of determining the extent and durability of the rain protection afforded by clothing fabrics has long been apparent. Although many laboratory tests are now in existence which can be performed with facility, no relationship has yet been established between the length of time a garment will keep the wearer dry and laboratory tests of the fabric from which that garment was made.

Obviously, the most direct means of determining the ability of a fabric to resist penetration is by testing in actual rainfall. This procedure is not feasible, however, because it is impossible to ensure that rainfall will occur at the desired time of test or that it will continue at a uniform rate for a sufficient period of time to permit direct comparisons between fabrics. Furthermore, it is difficult to obtain reliable test data under natural rain conditions because of the lack of control over such variables as wind velocity and air and water temperature.

The accurate reproduction of rain by artificial means which would provide the controls required for the establishment of a valid testing program was therefore undertaken by technologists of the Quartermaster Corps Research and Development Laboratories at Philadelphia [4]. As a result of this work, three nozzles were developed which were capable of producing artificial rain at the rates of 0.1, 1.0, and 3.0 inches per hour, duplicating with considerable accuracy the three prime characteristics of natural rainfall: intensity, drop size, and drop velocity.

These devices provided a unique testing instrument which was accepted as the most suitable criterion for determining the water-resistance of fabrics and garments and the efficacy of various repellent compounds. Two general methods of evaluation were used in the rainroom. One was the testing of garments worn by human subjects and the other the testing of fabrics on specially designed holders. Although tests on garments are desirable from the service standpoint, it has been found that the fabric holders can be used to furnish quantitative data of greater research value since by this method certain variables, such as the effect of seams and individual wearing characteristics of the human test subject, are eliminated.

The rainroom was recognized as a valid testing device, but its operation was costly and time-consuming and required much personnel. The study described in this report was therefore undertaken in an effort to establish a correlation between rainroom results and data obtained by the much simpler and more economical laboratory tests. Such a correlation would make it possible to predict the rainroom behavior of a given sample on the basis of laboratory tests and would thus eliminate the necessity for the

* Textile Series—Report No. 51. Released for public information by the Office of Technical Services, U. S. Department of Commerce.

TABLE I. CONSTRUCTIONAL FACTORS OF FABRICS IN SERIES I

Fabric No.	Weave	Weight (oz./sq.yd.)	Thickness (in.)	Texture Warp	Texture Filling	Yarn No. Warp	Yarn No. Filling
1	Poplin	6.20	.0156	107	54	40/2	15
2	Sateen	8.78	.0228	112	70	36/2	22/2
3	Oxford	8.50	.0191	132	46	36/2	13
4	Poplin	4.86	.0126	109	63	48/2	24
5	Poplin	4.39	.0142	117	42	33	15
6	Twill	5.21	.0161	118	55	22	25
7	Gabardine	9.01	.0287	133	75	28/2	21

rainroom, except possibly as a final check on the most desirable fabrics or fabric-repellent combinations. The study involved the establishment of two separate correlations: first, between rainroom results using the two different methods of testing—*viz.*, as garments worn on human test subjects and as swatches clamped on fabric holders; and second (provided a satisfactory relationship is found to exist in the first), between laboratory data and the results of rainroom tests using fabric holders.

Two series of fabrics in different ranges of water-resistance were used for this correlation study. The first (Series I) included several commercial- and military-type fabrics whose resistance to penetration varied from very poor to very good. The second (Series II) consisted of 25 experimental materials of excellent water-resistance which had been produced with controlled variations in constructional factors such as yarn twist, size, and ply; texture; and weight.

Although this study represents a relatively comprehensive correlation analysis between laboratory and rainroom tests, it should be emphasized that the number of fabrics in Series I (7) is not sufficient to cover completely the wide range of water-resistance represented. The 25 fabrics in Series II, on the other hand, all of which possess a high degree of water-resistance, should be a satisfactory number of samples, although here a limitation is present in that only one weave (Oxford) was tested. These data, however, if properly used, should give some valuable indications as to the protection which will be afforded by garments made from fabrics tested in the laboratory.

Experimental Procedure

Materials

Series I.—The 7 fabrics used in the first part of this study were civilian- and Army-type materials, finished, dyed, and treated with the same durable water-repellent compound. The physical properties of these fabrics are listed in Table I.

TABLE II. CONSTRUCTIONAL FACTORS OF FABRICS IN SERIES II

Fabric No.	Weight (oz./sq.yd.)	Thickness (in.)	Texture Warp	Texture Filling	Yarn No. Warp	Yarn No. Filling
1	5.89	.012	200	95	75.7/2	75.7/2
2	7.57	.015	161	79	75.7/3	75.7/3
3	8.75	.016	144	70	60.2/3	60.2/3
4	11.12	.021	116	56	38.3/3	38.3/3
5	15.78	.029	80	40	18.6/3	18.6/3
6	6.17	.013	196	83	75.7/2	57.9/2
7	7.77	.016	160	70	75.7/3	60.2/3
8	9.17	.018	151	60	60.2/3	43.3/3
9	16.29	.031	80	35	18.6/3	14.2/3
10	23.70	.045	56	24	9.1/3	7.0/3
11	6.84	.015	194	71	75.7/2	41.1/2
12	8.34	.018	159	56	75.7/3	38.3/3
13	9.85	.020	140	49	60.2/3	29.3/3
14	13.70	.029	98	34	29.3/3	14.2/3
15	25.26	.050	56	19	9.1/3	4.5/3
16	6.07	.013	185	89	75.7/2	57.9/2
17	7.71	.016	150	74	75.7/3	60.2/3
18	16.16	.030	75	36	18.6/3	14.2/3
19	13.26	.025	82	39	29.3/3	21.9/3
20	23.19	.045	47	21	9.1/3	4.5/3
21	7.89	.016	162	73	75.7/3	60.2/3
22	16.75	.031	79	36	18.6/3	14.2/3
23	7.26	.014	194	82	75.7/2	41.1/2
24	15.27	.027	98	40	29.3/3	14.2/3
25	19.07	.034	79	32	18.6/3	9.1/3

Series II.—The 25 fabrics used in the second part of this study were all experimental Oxford-weave fabrics ranging in weight from 6 to 25 oz./sq. yd. These fabrics were all finished alike and treated with an Army-approved, durable water-repellent compound. The physical properties of these fabrics are listed in Table II.

Rainroom Tests

Description of the rainroom has been previously given in an article by Landsberg and Moody [4].

Garments.—The fabrics in Series I were tested by Slowiński and Pope [6] in the form of single- and double-layer garments worn by human subjects, following (with some modification) the procedure for rainroom evaluation developed by the Quartermaster Research and Development Laboratories at Philadelphia. Each man wore his own suit coat, shirt, and undershirt under the raincoat. The raincoats were worn until serious penetration occurred or until a total wearing time of 2 hours had elapsed. Serious penetration was defined as (*a*) wetting of

the suit coat over an area of approximately 50 square inches, or (*b*) absorption by the suit coat to the extent of 30 or more grams. One to four single- or double-layer raincoats of each fabric in Series I were tested. No garment evaluations were made of the Series II fabrics.

Fabric Swatches.—In the evaluation of fabrics in the form of sample swatches, a special hemicylindrical metal holder approximately 14 inches in length and 18 inches in circumference was used (see Figure 1). The fabrics were placed on the holder and kept in position by a hinged clamp lined with sponge rubber. A uniform tension was maintained throughout the test by a 1-pound metal weight. Underneath the test fabric was placed an 8.2-ounce twill material to absorb the moisture penetrating the specimen. This backing fabric was removed at specific time intervals and weighed. The increase in weight of the twill cloth was used as a measure of fabric penetration. When 20 grams of water had penetrated the fabric being tested, it was considered to have failed and the time for this failure to occur was used as its "rainroom penetration time." All the fabrics in both series were evaluated in this manner.

FIG. 1. *Fabric holder used for evaluating samples in the rainroom.*

Laboratory Tests

There are essentially two types of laboratory water-resistance tests: those which measure resistance to the passage of water through a fabric, and those which measure resistance to wetting of the fabric itself. The present paper is limited to consideration of the first of these types—*i.e.*, penetration tests—for two reasons. First, in the case of water-repellent clothing, the ultimate criterion from the standpoint of protection provided must be the amount of water penetrating the fabric and not that which is merely absorbed or which wets the outer surface. Second, the tests which measure the resistance of the fabric to wetting are, for the most part, not indicative of the inherent water-resistance of its structure (as are the penetration tests) but are indicative of the efficacy of the water-repellent compound. Tests of this type, such as static- and dynamic-absorption and several surface-repellency tests, including spray-rating, contact-angle, and wetting-time determinations, were conducted, but since all the fabrics were treated in a uniform manner with the same repellent, the results fell within an exceedingly narrow range. Hence, the primary significance of these tests was to establish the uniformity of the treatment and to indicate that any differences in penetration test results could be attributed primarily to differences in fabric structure.

The penetration tests described in this paper are as follows: (1) drop penetration, (2) Suter hydrostatic pressure, (3) du Pont rain test, and (4) Bundesmann.

In these tests (with the exception of the hydrostatic) the amount and pressure of the water are kept constant and the relationship between time and quantity of water penetrating the material is determined. The end point is expressed either in terms of the amount of water collected in a given period of time or the time necessary to collect a specific amount of water. The penetration tests differ from each other mainly in the amount and the impact of the water striking the fabric. In evaluating fabrics of good water-resistance, differences are more clearly shown by tests in which the water strikes the sample with a relatively high velocity, whereas less severe conditions of test are required to show differences among the poorer fabrics.

The hydrostatic test differs from others in that (1) the pressure is increased at a controlled rate instead of being kept constant and (2) the fabric is maintained in constant contact with the water, producing in essence a static rather than a dynamic type of test.

A brief description of the tests follows:

(1) *Drop-Penetration Test.*—In this test [2] the water which penetrates the fabric specimen passes through a crescent-shaped slit in a fabric holder and is collected in a graduated cylinder. The drop-penetration value is reported as the time required to collect 10 ml. of water. All of the fabrics in Series I were evaluated in this manner. In the evaluation of the Series II fabrics, a slight modification was made in the testing procedure because of their very high water-resistance. The modification consisted of measuring the amount of water penetrating the test specimen during a 3-hour period.

(2) *Hydrostatic-Pressure Test.*—This test is also described in Federal Specification CCC-T-191a (Supplement 8, October, 1945) [2]. A hydrostatic head is built up on the face of the fabric at the rate of 1 cm. per second. The height measured at the moment of penetration of three drops of water is taken as the hydrostatic-pressure value for the fabric. The fabrics in both Series I and Series II were tested in the manner specified.

(3) *Du Pont Rain Test.*—This test is described in a published article by Slowinske and Pope [6]. A horizontal water spray from a specified type of nozzle is directed against the test fabric placed at right angles to the spray, 12 inches from the nozzle. The water penetrating a specimen is measured by weighing a blotter placed behind the fabric. The rain-test value may be expressed either as the time required for the penetration of a specified weight of water, or as the weight of water penetrating within a specified time. Another alternative procedure consists of determining the time required for the penetration of the first drop of water, as visually noted on the backing blotter. This test is applicable to a wide range of fabrics by virtue of the fact that the head of water can be varied from 2 to 8 feet, thus increasing or decreasing the impact with which the water strikes the fabrics. For the fabrics in Series I, no modification of the test instrument was necessary. In the evaluation of the water-resistant fabrics in Series II, three modifications were made: (1) the distance of the nozzle from the fabric was decreased to 6 inches, (2) the design of the apparatus was changed in order to ensure that the streams of water hitting the specimens would be parallel [3], and (3) the time of test was increased to 20 minutes.

(4) *Bundesmann Test.*—This test is described in Tentative Textile Standards Nos. 7 and 8 of the British Textile Institute [5]. Fabric specimens are mounted over a series of revolving cups and subjected to a controlled spray. The underside of each fabric is rubbed by a rotating wiper arm inside the cup. The resistance of the specimen to penetration is indicated by the amount of water collected in the cups in a given time. This test is standard in European countries and in Canada but has not been favorably accepted in this country because of the high cost of the apparatus required and the operational difficulties involved in the performance of the test. This test was used only in the evaluation of the fabrics in Series I.

Results and Discussion

Rainroom Correlations

Prior to the establishment of relationships between laboratory and rainroom test results, it was necessary to determine two correlations between different methods of testing in the rainroom. The first of these concerned the extent of agreement which existed between tests of garments worn by human subjects and tests of sample swatches attached to fabric holders. The second was a correlation between results obtained on the fabric holders under a 1-inch-per-hour rainfall and a 3-inch-per-hour rainfall.

The fabrics in Series I were tested in the rainroom both as garments worn by human subjects and as test swatches mounted on fabric holders. Single-layer garments and fabric specimens were subjected to a 1-inch-per-hour rainfall. For the single layers this intensity was found to be most suitable because it was sufficient to cause penetration through most of the materials in a relatively short time and yet did not produce such rapid failure that it was impossible to obtain an accurate ranking of the water-resistance of the specimens and/or garments. In double layers the fabrics were tested only in swatch form mounted in fabric holders under a 3-inch-per-hour rainfall. Although comparable double-layer garment data would have been desirable, it was not feasible to conduct a test with human subjects for

a period longer than 2 hours, in which time only the two poorest double-layer fabrics showed signs of failure.

A comparison of penetration time obtained for garments on human subjects and fabrics swatches on sample holders, as shown in Table III, indicates that under a 1-inch-per-hour rainfall both of these methods rank the fabrics in the same order: 3, 2, 1, 4, 7, 6, 5.

Figure 2 indicates relatively good agreement between data obtained with fabric in holders and with garments under a 1-inch-per-hour rainfall when these were tested in single layers. The correlation coefficient expressing the degree of relationship between these results was 0.80, based on 6 of the 7 fabrics. The Oxford fabric (No. 3) is not included because no failure was obtained on it in the garment form. The coefficient of correlation is significant but not as high as would have been desired. However, practical consideration precluded extended garment tests, making it necessary to accept results obtained on fabric holders as a basis for correlation with laboratory tests.

Since the correlation between tests of garments and fabric specimens on holders is considered satisfactory, it follows that fabric specimen data can be used to estimate the approximate performance under a 1-inch-per-hour rainfall of garments made from those fabrics. It also follows that the same data can be used for correlation purposes between rainroom and laboratory test results. This correlation must be supplemented, however, with a correlation between results obtained under a 1-inch-per-hour rainfall since this intensity is required for practical testing of double-layer fabrics. As shown in Figure 3, a high correlation coefficient of 0.97 was found for this relationship. The good agreement between the penetration time for a single-layer fabric under a 1-inch-per-hour rainfall and that for a double-layer fabric under a 3-inch-per-hour rainfall indicates that the increased intensity is compensated for by doubling the fabrics.

TABLE III. RAINROOM TEST RESULTS ON SERIES I FABRICS (TIME IN MINUTES FOR PENETRATION TO OCCUR)

Fabric No.	Fabric	Garments Single layer (1 in./hr. rainfall)	Fabric swatches Single layer (1 in./hr. rainfall)	Fabric swatches Double layer (3 in./hr. rainfall)
3	Oxford	120+	1,760.0*	1,680.0
2	Sateen	100	625.0*	1,530.0
1	Poplin	80	60.0	190.0
4	Poplin	60	37.5	60.0
7	Gabardine	25	30.0	52.5
6	Twill	20	22.5	26.3
5	Poplin	10	15.0	15.0

* Values estimated by extrapolation of curve showing rate of penetration after 6.5 hours.

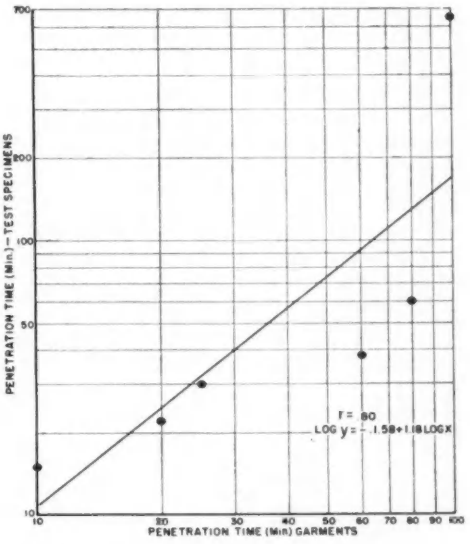

FIG. 2. *Relationship between average penetration time for garments and test specimens on fabric holders evaluated in the rainroom (1 in. hr. rainfall). (Series I—Single-layer fabrics and garments.)*

Correlation of Rainroom and Laboratory Tests

The rainroom evaluation of the fabrics and garments in Series I has been discussed. The 25 fabrics in Series II were evaluated on fabric holders under a 3-inch-per-hour simulated rainfall. The fabrics in this series possessed such superior water-resistance that it was not feasible to evaluate them under a 1-inch-per-hour rainfall so as to permit direct comparisons with the fabrics in Series I. Even under a 3-inch-per-hour rainfall some of the fabrics required nearly 100 hours of testing in order to obtain a break-through.

The four laboratory penetration tests ranked the fabrics in Series I in single- or double-layers nearly in the same order as did the fabric holders in the rainroom, as shown in Table IV. Because of the large number of fabrics in Series II, the establish-

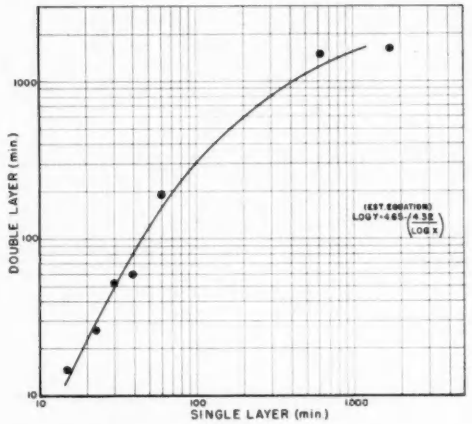

FIG. 3. *Relationship between rainroom penetration time of single layer (1 in./hr. rainfall) and double layer (3 in./hr. rainfall). (Series I fabrics.)*

ment of a rank correlation was not considered suitable. The data are summarized, however, in Table V.

A mathematical relationship to permit prediction of rain performance on the basis of data obtained with fabric specimens on holders (which correlate with garment performance) was established by calculating regression equations. A coefficient of correlation was used to express the closeness of the relationships. In addition to the regression equations and coefficients of correlation, the standard error of estimates and 90-percent confidence limits, shown in Table VI, were also calculated to permit more valid interpretation of the data [1].

The data shown in Tables IV and V have been plotted to show the relationships between the water-resistance of the fabrics in Series I, as measured in the rainroom and as determined by the drop-penetration (Figure 4), Bundesmann (Figure 5), du Pont (two methods, Figures 6 and 7), and hydrostatic-pressure (Figure 8) tests. Data for both single and double layers of the fabrics are plotted in all the graphs except that of Figure 7, in which case the conditions of test precluded evaluation in double-layer form. Similar graphs have also been plotted for the Series II fabrics (single layers only), based on the results shown in Table V, to demonstrate the relationships between rainroom data and the values obtained by the drop-penetration (Figure 9), du Pont (Figure 10), and hydrostatic-pressure (Figure 11) tests.

The graphs showing the relationships between rainroom and laboratory tests on the fabrics in both Series I and Series II covered such a wide range (the best fabric often numerically exceeding the worst by a ratio of more than 1,000 to 1) that it was found expedient to plot both laboratory and rainroom values logarithmically. When this was done, in most cases a straight line could be drawn reasonably close to the plotted points. The relationship between the results of a given laboratory test and the rainroom penetration time can be represented by the equation:

$$\text{Log } Y = a + b \text{ log } X,$$

where Y and X represent rainroom and laboratory test values, respectively. Because the hydrostatic test values for the 7 fabrics in Series I were found to vary within a comparatively narrow range, they could be adequately plotted along the arithmetic scale of semi-logarithmic paper, with the rainroom data plotted along the logarithmic scale. The equation derived in this case was

$$\text{Log } Y = a + b X.$$

Inspection of these graphs reveals that there is often a considerable discrepancy between the estimated and actual rainroom values. Thus in Figure 4, for example, it can be seen that a single-layer fabric in Series I with a drop-penetration time of 8.3 minutes has an estimated penetration time under a 1-inch-per-hour rainfall of about 240 minutes, whereas the fabric actually held up for 625 minutes. Many points which deviate similarly from the regression line can be noted in all of the graphs for both Series I and Series II.

The deviations of the actual from the estimated values are not, in general, attributable to lack of precision in the various laboratory test methods or in the rainroom, since in most cases each point on the graphs represents the average of a number of individual determinations which are in relatively good agreement among themselves. The deviations appear to arise from the fact that each test which was correlated with the rainroom does not necessarily measure exactly the same characteristics as the rainroom. Since the fabrics tested varied widely in type of weave, weight, yarn number, twist, etc., it can readily be seen that unless each of the laboratory tests is affected by these various characteristics

TABLE IV. COMPARISON OF RAINROOM AND LABORATORY TEST RESULTS ON SERIES I FABRICS

Fabric No. and Weave	Rainroom* (min.)	Hydrostatic (cm.)	Drop penetration (min./10 ml.)	Bundesmann (g./20 min.)	du Pont rain test 2 feet (g./5 min.)	du Pont rain test 3 feet (sec. for initial penetration)	du Pont rain test 3 feet (sec./4 g.)	du Pont rain test 8 feet (sec. for initial penetration)
Single fabrics								
3 Oxford	1,760.0†	63	54.0	0.0	0.0	930	144,000+‡	
2 Sateen	625.0†	40	8.3	0.2	0.1	116	4,100§	
1 Poplin	60.0	36	3.8	1.8	0.2	20	1,500	
4 Poplin	37.5	34	3.8	3.9	2.2	8	120	
7 Gabardine	30.0	26	1.4	20.0	11.7	1	14	
6 Twill	22.5	26	0.8	59.0	12.7	1	4	
5 Poplin	15.0	18	0.75	99.0	13.0	1	2	
Double fabrics								
3 Oxford	1,680.0	88	300+	0.0				18,000+
2 Sateen	1,530.0	52	91.0	0.0				—
1 Poplin	190.0	48	18.0	0.0				1,116.0
4 Poplin	60.0	42	21.0	0.2				38.0
7 Gabardine	52.5	30	9.2	0.1				35.0
6 Twill	26.3	29	2.0	0.3				7.0
5 Poplin	15.0	21	1.3	3.0				3.5

* For single fabrics, 1 in./hr.; for double fabrics, 3 in./hr.
† Values estimated by extrapolation of curve showing rate of penetration after 6.5 hrs.
‡ Value estimated by extrapolation; <0.1 g. after 3,600 sec.
§ Value estimated by extrapolation; 3.5 g. after 3,600 sec.

TABLE V. RAINROOM AND LABORATORY RESULTS ON SERIES II FABRICS

Fabric No.	Rainroom penetration (time in hrs.)	Drop penetration (ml./3 hrs.)	Hydrostatic pressure (cm.)	Rain tester Initial penetration (time in sec.)	Rain tester (g./20 min.)
1	4.5	44	63	19	.68
2	11.1	12	65	18	.34
3	21.7	13	68	35	.55
4	44.5	3	75	48	.11
5	39.2	3	67	79	.27
6	5.0	34	69	13	.40
7	23.1	19	72	50	.56
8	20.3	9	66	56	.49
9	62.7	3	67	92	.26
10	60.9	5	56	209	.19
11	15.3	24	70	22	.74
12	25.8	17	70	50	.63
13	30.4	12	69	71	.46
14	55.5	5	58	25	.83
15	83.6	1	55	66	.13
16	12.3	31	65	28	.78
17	27.1	17	71	113	.30
18	37.5	2	62	127	.19
19	27.2	15	48	7	3.01
20	95.3	2	36	9	2.33
21	24.0	11	82	735	.17
22	92.5	4	70	198	.06
23	34.9	5	91	625	.22
24	37.0	5	80	1,017	.08
25	98.1	2	66	756	.07

TABLE VI. STATISTICAL ANALYSIS OF CORRELATIONS OF LABORATORY AND RAINROOM TEST RESULTS

Test	Regression equation (X = Laboratory value, Y = Rainroom value)	Correlation coefficient	Standard error of estimate (log value)	90% confidence limits (% of estimated Y values) Lower	Upper
Series I					
Single layers					
Drop penetration (min./10 ml.)	$\log Y = 1.30 + 1.15 \log X$	0.94	0.28	52	192
Hydrostatic (cm.)	$\log Y = 0.131 + .052X$	0.93	0.33	47	214
du Pont rain tester					
3-ft. head, initial penetration (sec.)	$\log Y = 1.24 + 0.66 \log X$	0.96	0.24	57	175
3-ft. head (sec./4 g.)	$\log Y = 0.96 + 0.416 \log X$	0.94	0.31	49	204
2-ft. head (g./5 min.)	$\log Y = 1.86 - 0.523 \log X$	−0.86	0.33	47	214
Bundesmann (g./20 min.)	$\log Y = 2.15 - 0.53 \log X$	−0.93	0.24	57	175
Double layers					
Drop penetration (min./10 ml.)	$\log Y = 1.01 + 0.92 \log X$	0.95	0.28	52	192
Hydrostatic (cm.)	$\log Y = 0.43 + .053X$	0.90	0.34	46	218
du Pont rain tester					
8-ft. head, initial penetration (sec.)	$\log Y = 0.93 + 0.512 \log X$	0.99	0.13	74	135
Bundesmann (g./20 min.)	$\log Y = 1.35 - 0.39 \log X$	−0.90	0.15	71	141
Series II					
Single layers					
Drop penetration (ml./3 hrs.)	$\log Y = 2.09 - 0.69 \log X$	−0.87	0.18	66	150
Hydrostatic (cm.)	(No correlation)	—	—	—	—
du Pont rain tester					
8-ft. head (g./20 min.)	$\log Y = 1.34 - 0.302 \log X$	−0.36	0.34	46	218
8-ft. head, initial penetration (sec.)	$\log Y = 0.981 + 0.271 \log X$	0.44	0.33	47	214

in exactly the same way as is the rainroom, some discrepancies must be expected between actual and estimated values. For example, fabric thickness may have a much greater influence on rainroom than on drop-penetration test results because of the fact that the former is not an accelerated test and is less severe in its action than the latter. Thus, many of the points on the charts are some distance from the regression line, indicating that the rainroom values estimated on the basis of laboratory test results are not exactly the same as the actual results.

For this reason, each graph or estimating equation indicates not the exact but the most probable rainroom penetration time which would be obtained for a fabric of a given water-resistance as measured by the laboratory test in question. Since this is the case, a standard error of estimate was calculated to permit prediction of the probability that the rainroom time would fall within a certain range of values.

To provide a margin of safety for estimating rainroom values, the standard error of estimate was used to calculate the 90-percent confidence limits [1] for each test. These limits are expressed in Table VI in terms of percentages of the estimated Y or rainroom value. The confidence limits permit estimation of the range within which rainroom values could be expected to fall nine out of ten times on the basis of the laboratory test value. For example, a single-layer fabric with a drop-penetration value of 1.4 minutes resists penetration in the rainroom under a 1-inch-per-hour rainfall for approximately 30 minutes, according to Figure 4. Using the values for the 90-percent limits (lower limit, 52 per cent; upper limit, 191 percent), it can be seen that the rainroom penetration time would be no less than 15.6 and no greater than 57.3 minutes nine times out of ten.

In a like manner the time limits within which any specimen would be expected to resist penetration in the rainroom can be determined.

The information obtained by this correlation study can be put to practical use in establishing minimum standards for laboratory test values. For example, it is desired to ascertain whether fabrics which will eventually be made into garments will withstand a 1-inch-per-hour rainfall for 100 minutes. According to Figure 4, the fabric whose most probable rainroom time is 100 minutes has a drop-penetration time of 4.0 minutes. However, the 90-percent confidence limits are such that a fabric with a drop-penetration time of 4.0 minutes could resist penetration in the rainroom for as short a period of time as 52 minutes. Therefore, the drop-penetration value must be set higher in order to provide a margin of safety. Since the desired minimum value is 100 minutes and the lower limit for the drop-penetration test in single layers is 52 percent (Table VI), it can be seen that by dividing 100 minutes by 52 percent a value of 192 minutes is obtained. This value is therefore the one whose lower confidence limit is 100 minutes. Using Figure 4 again, it can be seen that a fabric with a rainroom penetration time of 192 minutes will have a drop-penetration time of approximately 75 minutes. This value is therefore chosen as the minimum drop-penetration value which will ensure with reasonable certainty a rainroom penetration time of at least 100 minutes. The same procedure can be applied to the other laboratory tests to obtain any desired minimum rainroom value.

Having shown how the laboratory penetration tests can be used to predict rain performance, it became desirable to ascertain which of these gave the most reliable estimate of rain protection. The coefficients of correlation and the standard errors of estimate obtained for the tests used to evaluate the single-layer fabrics in Series I were nearly the same. The closeness of these two statistical criteria indicates that, for all practical purposes, no one of these tests appears to predict the rain performance of garments better than another. However, in the evaluation of the double-layer fabrics in Series I the du Pont rain test (8-foot head), with a coefficient of correlation of 0.99 and a standard error of estimate of 0.13, appears to be more suitable than the other laboratory tests for predicting rainroom penetration time. The data obtained on this series of fabrics for the drop-penetration, hydrostatic, and Bundesmann tests showed that no one of these tests was markedly superior to the others. The Bundesmann test, because of its low standard error of estimate, appears to be somewhat better than the others, but the small number of fabrics on which data could be obtained (4 fabrics) did not permit this test to be classified as superior to the other two.

In a like manner the efficacy of the drop-penetration test, the hydrostatic-pressure test, and the two modifications of the du Pont rain test, was determined, using the fabrics in Series II, which were evaluated in single layers under a 3-inch-per-hour rainfall. The first modification of the du Pont test consisted of weighing the amount of water which had penetrated the fabric in a 20-minute period. The second and more rapid method consisted of determining the time in seconds required for initial penetration (appearance of the first drop) to occur. The coefficients of correlation for the two values were -0.36 and 0.44, respectively, indicating only a slight degree of correlation between the du Pont test and the rainroom test for this series of fabrics. In this series, as shown in Figure 11, no correlation was observed between the hydrostatic-pressure test and rainroom penetration time. The drop-penetration test, which had proved to be satisfactory in the evaluation of single and double layers in Series I, also showed a high coefficient of correlation of -0.87 and a low standard error of estimate of 0.18 for the Series II fabrics, indicating that this test should be suitable for estimating rainroom penetration time.

The analyses made to this point on the fabrics in Series I appear to show that the four penetration tests can be used interchangeably to estimate rainroom protection. However, such is not the case with the hydrostatic-pressure test, in spite of the rather high coefficient of correlation and the relatively low standard error of estimate. For example, it can be seen in Figure 8 that a single-layer fabric with a hydrostatic-pressure value of 40 cm. gives an estimated rainroom penetration time of 160 minutes, and that a fabric with a hydrostatic-pressure value of 44 cm. gives an estimated rainroom value of 260 minutes. Therefore, a 10-percent increase in the average hydrostatic-pressure value corresponds to a 62.5-percent increase in rainroom penetration time. This percentage will increase at the higher hydrostatic values and decrease at the lower values. Since most good water-resistant fabrics have relatively high hydrostatic-pressure values, it can be

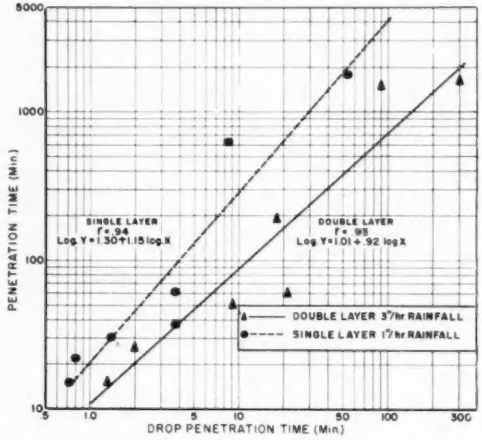

FIG. 4. *Relationship between penetration time (rainroom) and drop penetration for 7 single- and double-layer fabrics. (Series I fabrics.)*

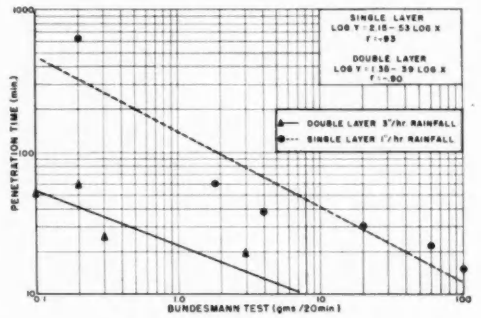

FIG. 5. *Correlation of rainroom data with Bundesmann test. (Series I fabrics.)*

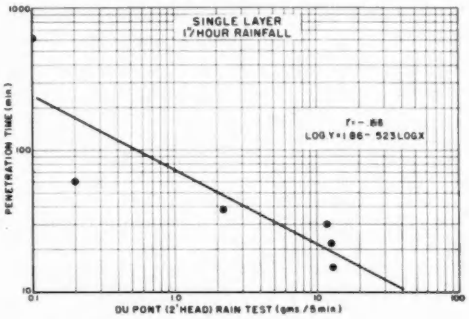

FIG. 7. *Correlation of rainroom data with du Pont rain test. (Series I fabrics.)*

seen that small percentage differences at these higher hydrostatic pressures will be reflected in large differences in rainroom penetration times. It can also be seen that it is entirely possible that several fabrics having nearly the same hydrostatic-pressure value could possess widely different protective values in the rainroom. The insensitivity of this laboratory test possibly explains the lack of correlation between the hydrostatic-pressure value and the rainroom penetration time on the Series II fabrics (Figure 11).

The du Pont and drop-penetration values are more reliable indicators of rainroom protection time than the hydrostatic-pressure values because given percentage changes in the two former tests are reflected in more similar percentage changes in rainroom values. For example, in the drop-penetration test (Figure 4), a 10-percent increase from 20 to 22 minutes corresponds to a 12.5-percent increase in rainroom protection time, and a 20-percent drop-

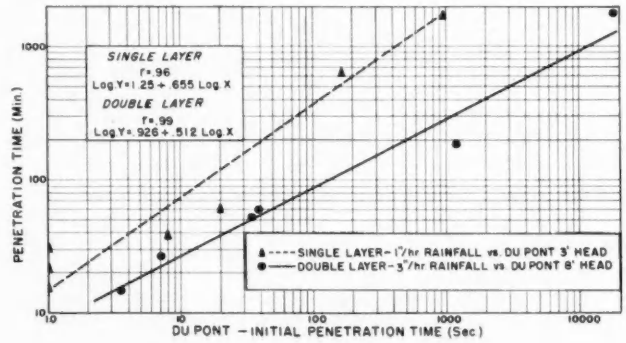
PENETRATION TIME (Min.)
DU PONT – INITIAL PENETRATION TIME (Sec)
SINGLE LAYER
r=.96
Log Y=1.25+.655 Log X
DOUBLE LAYER
r=.99
Log Y=.926+.512 Log X
SINGLE LAYER–1"/hr RAINFALL vs. DU PONT 3" HEAD
DOUBLE LAYER–3"/hr RAINFALL vs. DU PONT 8" HEAD

FIG. 6 *Correlation of rainroom data with du Pont rain test. (Series I fabrics.)*

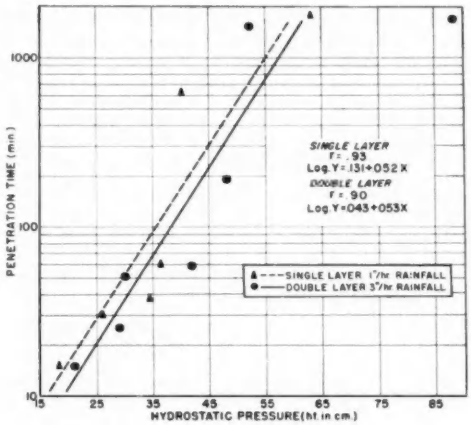

FIG. 8. *Correlation of rainroom data with hydrostatic-pressure test. (Series I fabrics.)*

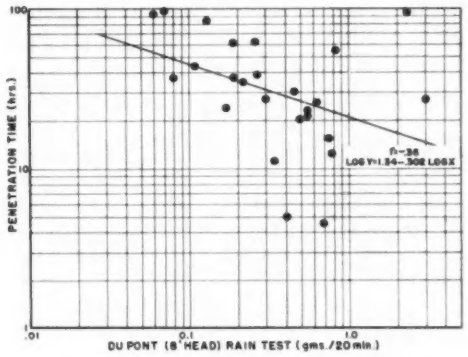

FIG. 10. *Correlation of rainroom data (3 in./hr. rainfall) with du Pont rain test for single layers of fabric. (Series II.)*

penetration increase from 20 to 24 minutes is the equivalent of an 18.7-percent rise in the rainroom value. In the case of the du Pont rain tester (Figure 6), as the laboratory value increases 10 or 20 percent from 20 seconds to 22 or 24 seconds, the corresponding rainroom change is 5.9, or 11.1 percent.

Based on the analyses which have been performed it has been shown that the drop-penetration test is most suitable for predicting rain performance of a wide range of fabrics. The du Pont rain test, although not as widely applicable as the drop-penetration test, is most suitable for estimating the rain performance of either single- or double-layer fabrics possessing the water-resistance characteristics desired by manufacturers producing civilian-type raincoats. The Bundesmann test appears to be almost as satisfactory as the rain tester but its limited use and range preclude its general acceptance. The hydrostatic-pressure test is not as suitable as the other penetration tests for predicting the rain performance of a wide range of fabrics. This may

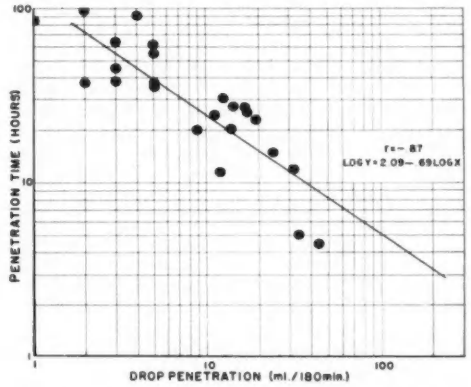

FIG. 9. *Correlation of rainroom data (3 in./hr.) with drop-penetration test for single layers of fabric. (Series II.)*

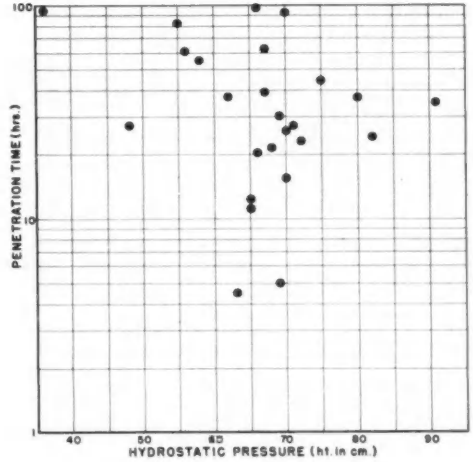

FIG. 11. *Correlation of rainroom data (3 in./hr. rainfall) with hydrostatic-pressure test for single layers of fabric. (Series II.)*

be due to its relative insensitivity to those characteristics which influence rain protection, such as thickness and density.

Conclusions

1. Data obtained using fabric holders correlate quite well with data obtained from garment wearing tests. Specimens tested on holders may be used in lieu of garments on human subjects to estimate the approximate protection afforded by fabrics under a specified rainfall.

2. The most probable rainroom protection time of a fabric treated with a durable water-repellent compound can be estimated from the results of laboratory penetration tests by use of the equations: Log $Y = a + b \log X$ for the du Pont, drop-penetration, and Bundesmann tests; and Log $Y = a + bX$ for the hydrostatic test.

3. The drop-penetration, Bundesmann, and du Pont tests are equally reliable for estimating the rainroom penetration time of single and double layers of fabric which resist penetration under a 1- and 3-inch rainfall, respectively, for a few minutes to approximately 24 hours. The complexity of the Bundesmann apparatus and its relatively limited range preclude its general acceptance as a test instrument.

4. The hydrostatic-pressure test correlates well within this range of water-resistance, but is apparently less sensitive to factors which influence rain protection than the other tests, and hence appears to be inherently less reliable.

5. For fabrics possessing a very high degree of water-resistance (5 to 100 hours for single layers under a 3-inch-per-hour rainfall) the drop-penetration test appears to be the only method which can be used to estimate rainroom penetration. Within this range of water-resistance the du Pont rain test is a poor index of rain protection and the hydrostatic test shows no correlation with the rainroom data.

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Literature Cited

1. Croxton, F. E., and Cowden, D. J., "Applied General Statistics," New York, Prentice-Hall Publishing Co., 1943, pp. 694–9.
2. Federal Specifications for Textiles; General Specifications, Test Methods, CCC–T–191a, Supplement 8, Oct. 8, 1945.
3. Institute of Textile Technology, Improvement of Water-Resistance of Cotton Military Fabrics (unpublished data).
4. Landsberg, M. L., and Moody, F. J., Devices for Producing Artificial Rainfall for Tests of Water-Repellent and Waterproof Materials, TEXTILE RESEARCH JOURNAL **16**, 211–18 (May 1946).
5. Lawrie, L. G., Tentative Textile Standards Nos. 7 and 8, *J. Text. Inst.* **33**, S49–52 (Oct. 1942); **38**, S1–14 (Feb. 1947).
6. Slowinske, G. A., and Pope, A. G., A Correlation of Water-Repellent Garment Performance and Laboratory Penetration Tests on Fabrics, *Am. Dyestuff Reptr.* **36**, 108–21 (Mar. 10, 1947).

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Understanding Accomplishment*

E. R. Schwarz†

"Considering the fact that he has been at it for only a few thousand years, man makes rather good textiles. He even is able to make acceptable textile fibers. Compared to nature, however, he still is an amateur with much to learn. Of course nature has carried on its development over some hundreds of thousands of years, and man cannot wait that long. Fortunately he doesn't have to. He can accomplish in a decade results that would require hundreds of years by empirical methods or thousands of years by processes of evolution and natural selection. This he can do in one way only: by research, intelligent and intensive research vigorously supported and patiently followed up in production. . . . In our modern tempo that industry is in danger which is in a static state. Research, with its yield of new and better products, is not a luxury; it is a necessity, often as a defense against encroachment from without. The world in which we live is going to change, politically and sociologically, no doubt, but also technically. We have no choice as to whether we will change with it. We simply have a choice as to whether we will change rapidly enough and sanely enough to remain part of the essential scheme of things."—Vannevar Bush

THERE are certain phrases in common usage which are of significance in our daily speech. One of these is "*the long and short of it.*" Perhaps its application to the textile industry will not be apparent at once—but a little consideration makes its pertinence clear. The manufacture of yarns by spinning certainly involves the conversion of relatively short fibers into long strands. And even the production of rayon or nylon involves combinations of relatively short molecular chains into long, continuous filaments. To complete the picture, it is also apparent that the formation of the long-chain molecules themselves is also a process of making long units out of short ones—a process which the chemist calls polymerization. For my purpose, the long and short of it is that we do not yet understand what we have accomplished.

For thousands of years fibers have been spun into yarns successfully enough to make possible beautiful and serviceable fabrics. These fabrics for centuries have had significance as a mirror of our progressing culture and civilization. Sacred vestiments, military uniforms, regal robes, legal gowns—all have been with us everywhere in the world since history began. Tapestries have pictured feats of arms, sports, industries, and personalities, and they show for each age a cross section of its life and times. Over the years, we have accomplished much by reason of textiles. But all of this development from remote antiquity to the present has been carried out without a real understanding of the process. In the spinning of yarns there has certainly been accomplishment; but it has been accomplishment achieved by trial and error and by imitation. It is much like our own accomplishment in the field of speech. We learn to speak as children by imitation of our elders, even to the extent of acquiring their particular accent. We do not have to be familiar with the technology of speech as exemplified in grammar, syntax, and diction. That comes later and serves at least two purposes: first, to improve our speech, and second, to aid us in new or unfamiliar circumstances, where imitation is impossible or where we must rely on our own ability.

Similarly in textiles, the industry has grown up by trial and error and by imitation. How often do we hear the explanation, "it always has been done that way!" Surely, fibers have always been twisted into yarns. We know what has been done, but not much about how it was done, and unfortunately we also know too little about why it was done.

Modern chemistry and physics have begun to show us a picture of what goes on inside the textile fibers we have been using for years. New words

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† In charge, Textile Division, Massachusetts Institute of Technology.

have become a part of the textile scientific literature. For instance, as a result of work in x-ray diffraction we have the concept of the *crystallite*, a repeating (geometric) unit of atomic and molecular structure, comparable in this respect to the repeat of a weave in a fabric. From work in the load-deformation characteristics of fibers, together with observations with the polarizing microscope, there comes the concept of the *micelle*, or a combination of long-chain molecules, with a behavior of its own. Such concepts aid greatly in explaining the swelling of fibers which takes place in finishing and the behavior of textile materials in service. They are not yet completely clear in the minds of research workers and are too often confused with each other. Study of these concepts should be increased and made a part of basic textile education. They are as important to a fiber as the fiber is to a yarn or as yarn is to fabric.

It will be agreed readily that short fibers are held in place in long yarns by friction and by reason of their curvature into helices. Friction can occur only when fibers touch each other. How many such points of contact are there per inch of yarn, and where are they? What is the nature of the surfaces of the fibers where they touch and how big is the area of contact? What is the normal pressure between surfaces at these contacts? To these questions we have no answers, yet they are fundamental to an understanding of yarn structure and behavior. We have accomplished a spun yarn, but we do not understand it.

A single yarn is composed of fibers, each of which is deformed into a helix. A plied yarn is similarly composed of single yarns and a cord is composed of plied yarns. Thus the axis of the single yarn is a helix which has been generated about a helix which is itself a helix with respect to the cord axis.

In use, the cord will be stressed along its axis, but this axis is merely a theoretical abstraction. It is just a dot-dash line in space. It is not composed of fiber substance, yet the cord withstands the stress. It does so because there are fibers at various distances from the imaginary axis and at various angles with it. All of these share the stresses among themselves in fashions too complex for analysis. They are not straight and they possess varying degrees of three-dimensional curvature. Geometrically, how are the individual fibers arranged with respect to the axis of the cord? How will they behave when the cord is stretched? The individual fibers are the working units of structure much like the members in a girder in a building, but we are not yet able to locate them as the architect can do with the steel in his building plans.

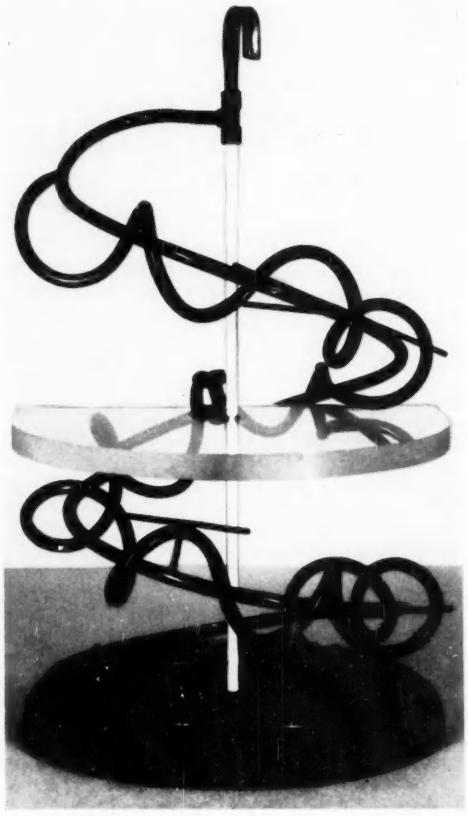

FIG. 1. *Model showing the geometry of cord structure, including cord, ply, and single axes, certain generators, and transverses essential to the understanding of twist relationships.*

We know that the lengths of the fibers composing a spun yarn are important. Should they be all of the same length, and, if so, what length? Should they be of different lengths, and, if so, what statistical distribution of lengths? Yes, we continue to make yarns as did our ancesters for countless generations, but do we understand what we have done? In the absence of such understanding do we have a satisfactory background upon which to base improve-

ments in yarns as to uniformity and serviceability? If not, we should be training our textile technologists so that they will have the proper information.

All this calls for an educational program which goes further than teaching mechanism, settings, speeds, drafts, and machine operation. Such a program should not omit information as to good practice. It should include basic information as to principles of operation of equipment and as to the structure and behavior of fibers of all kinds. In addition to the consideration of mechanical elements for handling and controlling fibers, such teaching should increasingly concern itself with electricity, aerodynamics, physical chemistry, and applied mathematics.

It is beginning to be realized that, in all, probability, much of the real action taking place in a cotton card, for example, is due to the forces produced on the fibers by their interaction with air. Much work has been done by scientists in studying the behavior of variously shaped surfaces in air and water streams. Work with wind tunnels and fluid streams and jets has produced our modern aviation and the modern turbine. If such research methods were reversed to provide information as to the control of moving air by means of properly designed surfaces we could better understand many of our present processing techniques and could look for new methods of fiber handling and control.

We should someday be able to place each individual fiber exactly where we want it in the yarn. But as yet we do not know exactly where we do want it or how to move it positively and with precision. We are dealing more and more intelligently with *groups* of fibers and we are learning slowly how to better control the measurements of aggregates of short fibers when they are combined with longer ones. Conventional drafting, however, continues to produce irregular yarns, and we have yet to state adequately what a uniform yarn really is. It is not enough to have only slight strength variation along a yarn. If, for instance, there are still thick and thin places and variations in twist distribution, the yarn cannot be considered really uniform.

Textile education should involve a thorough training in fundamental operations, whatever the fiber being handled. The student should be aware early of what the over-all problem is and should then be taught how to select and combine the essential operations so that any fiber or combination of fibers could be processed most efficiently. These might well include grading, sorting, opening, mixing, blending, cleaning, paralleling, evening, attenuating, twisting, and packaging.

Certain machines combine several of these operations, as in carding, where there is opening, blending, cleaning, and attenuation; or as in a slubber, where there is evening, attenuation, twisting, and packaging. With the increased use of man-made fibers, alone or in combination with other fibers, proper selection of operations and of the machines employing them is important. In this connection, departures from conventional processes are increasing. Nor is this line of development confined to other than natural fibers or to blends, since worsted yarn is now being manufactured by the so-called "American system."

Yarns and fabrics are being "engineered" more and more to meet requirements of service, and here the behavior of the fibers involved merits serious consideration. It does not necessarily follow that the materials we have been producing are optimum for certain specific applications. It therefore becomes necessary to study how the usefulness of the cloth can be measured and then how the proper fibers and construction of yarn and fabric can be discovered. We still cannot do as the engineer does when he specifies the materials and structural combinations of them for a particular bridge and then is sure that the bridge will function as planned. Our information as to the load-deformation characteristics of fibers is still fragmentary. Textile research is showing us that fibers possess a memory; that they behave not only in accordance with what is presently done to them, but also in accord with their previous history. They possess varying degrees of ability to recover after deformation. They are affected by temperature, rate of load application, rate of deformation, and moisture regain. Test data therefore are best interpreted by mathematical analysis based upon the theory of probability and involving correlation studies, the analysis of variance, significance determinations, and quality-control techniques.

In so simple a laboratory determination as that of the twist in a yarn, there may be variation in turns per inch from inside to outside layers in a bobbin, from bobbin to bobbin on a given spindle, from spindle to spindle in a machine, and from machine to machine in a section of the mill. If samples are properly taken and twist tests are properly carried

out, it becomes possible to find out, by an analysis of variance, how much each source of variation contributes to the whole. Furthermore, it can be shown whether or not these variations are of real significance and whether they are interrelated. Reasons for the variations produced may then be established and investigated and sound improvements may be made. None of this can be done without a real understanding of the principles basic to this branch of statistics. For control of quality in a mill, such preliminary work as this must be undertaken before the control chart can be constructed intelligently, much less used.

The result of research, therefore, is not the data but *the intelligent interpretation* of the data. Variations may confuse and conceal the real story of what is happening unless they are properly evaluated and analyzed. Properly planned sampling and techniques of analysis are vital to correct conclusions.

Textile education must provide a background of knowledge of physics, chemistry, biology, mathematics, and properties of materials. Not only is this needed for the research worker but for the executive as well. More and more often the executive must base decisions upon technical information coming to him from laboratories. This must be done intelligently. Experience indicates that the individual with basic scientific or engineering knowledge can acquire details of textile technology rapidly and easily. On the contrary, the individual with long experience in textile manufacturing finds it difficult or impossible to acquire basic scientific knowledge. Adult education is slow and adult re-education is even slower. The textile school's curriculum must therefore include additional mathematics and science. Training must be done in the early years of the technologist's development, and he must have a working background of information upon which he can draw intensively and intelligently throughout the remainder of his career.

For a successful textile education in these days, emphasis should be placed upon principles and upon training in analytical thinking rather than predominantly upon details of practice and the memorizing of settings and of formulas. Emphasis should be placed not so much upon accomplishment as upon understanding of what is being done. Imitation may be the sincerest form of flattery—but such flattery now in the textile industry and in textile education is a form of self-delusion. That our future may be secure, our educational program must seek to train for understanding of our accomplishments.

Through countless centuries have spindles whirled
  And danced beneath the spinner's skillful hands;
Combining from the corners of the world
A myriad of fibers into strands.
  Each filament alone, a tiny part,
But still essential. All linked each to each
To form a cloth which legend from the start
  Of history has loomed for us, to teach
In pictured tapestry how man may live
With man. Each yarn a life line spun by fate
  To measure out his destiny and give
A sign of his accomplishment and great
Achievement as an artisan. His hand
  Has shown his skill and now his brain
Must seek to answer and to understand
The mystery of why these threads contain
  Within their ordered structure, in each turn,
Ability to render service still
To those who follow. Seek the truth and learn
  The secrets of your hard-earned skill!—E. R. S.

(*Manuscript received June 7, 1949.*)

ABSTRACTS

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A list of periodicals, the abbreviations used in reference to articles, and the addresses of publishers of English-language publications is given in the January, 1949, issue, page 46.

ANALYSIS: TESTING: LABORATORY METHODS

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Elasticity and Viscosity

Measurements of shear elasticity and viscosity of liquids at ultrasonic frequencies. W. P. Mason, W. O. Baker, H. J. McSkimin, and J. H. Heiss. *Phys. Rev.* **75**, 936–46 (Mar. 1949).

Shear elastic and viscous effects exhibited by long-chain polymer liquids when sheared at various frequencies are discussed. A method is described for extending the range of shear wave measurements from a maximum frequency of 3×10^8 cycles obtained with a torsionally vibrating crystal to 60 megacycles by observing the effect on a series of shear waves in a fused quartz rod of terminating the rod by a thin layer of liquid. Both techniques are applied in a study of polyisobutylene and poly-α-methyl styrene liquids. L. P. Witnauer

Text. Research J. Aug. 1949

Fiber Mounting Medium

An improved mounting medium for textile fibers. H. H. Broadfoot and E. R. Schwarz. TEXTILE RESEARCH JOURNAL **18**, 756–8 (Dec. 1948).

A technique for mounting textile fibers on the microscope slide is described. The mounting medium used in this report, which has a refractive index that is approximately halfway between the maximum and minimum indices of the common fibers, gives good structure details of the fiber. By varying the composition of the mixture, it is possible to adjust the refractive index to a value most suitable for a particular fiber.

Text. Research J. Aug. 1949

Determination of Nitrophenols in Felt

Determination of some nitrophenolic proofing agents in felt. J. G. Norvall and C. Kenyon. *Analyst* **73**, 551–4 (1948) (through *Chem. Abstr.* **43**, 1194*a* (Feb. 10, 1949)).

A new method is described for determining *p*-nitrophenol and 2,4-dinitro-1-naphthol or *p*-nitrophenol and 4,6-dinitro-*o*-cresol in felt that has been treated to give protection against bacterial and fungicidal action. The results are within 3% accuracy. The nitro compounds are extracted from the felt with hot water, dissolved in Et_2O, and separated by chromatography. Then the individual constituents are determined by colorimetry. The method was tested with synthetic mixtures of compositions unknown to the analyst.

Text. Research J. Aug. 1949

Fractionation of High Polymers

The use of metal membranes for fractionating high polymers. J. L. Rosenberg and C. O. Beckmann. *J. Colloid Sci.* **3**, 483–503 (Dec. 1948).

A diffusion cell has been constructed with highly permeable stainless-steel membranes. The cell allows more rapid diffusion than previously described glass-membraned cells. The method of gaseous diffusion separation has been modified for use in the fractionation of high polymers in solution. Experiments have been performed to demonstrate the partial fractionation of mixtures. A method has been proposed for the construction of size-distribution curves by combining data from free diffusion measurements on the fractions obtained by membrane diffusion. Advantages of the diffusion method over the fractional precipitation method are predicted for cases where very large molecular-weight fractions free of low-molecular-weight material are desired. Authors

Text. Research J. Aug. 1949

Sedimentation Constant

Concentration dependence of sedimentation constant for nitrocellulose. I. Julliander. *J. Polymer Sci.* **3**, 631–3 (Oct. 1948).

An empirical method of correcting the sedimentation constant of high

polymers for its concentration dependence is proposed. It should be possible to determine the sedimentation constant at infinite dilution with reasonable accuracy from experiments at concentrations where reliable measurements can be made.
Text. Research J. Aug. 1949 Author

CHEMICAL and PHYSICAL RESEARCH

★

Polymerization of Cellulose in Cotton

Degree of polymerization of cellulose in cotton fibers. Lyle E. Hessler, George V. Merola, and Earl E. Berkley. TEXTILE RESEARCH JOURNAL **18**, 628–34 (Oct. 1948).

Cotton fiber from unopened bolls gave values for degrees of polymerization (D.P.) of 5,940 for primary walls only and 10,650 for fibers with secondary thickening. The young fiber with primary walls only was more sensitive to degradation both when dried without treatment and when the moisture was replaced by organic solvents. The greatest cellulose breakdown occurred when the fiber was oven-dried at temperatures of 105°C and above. The fibers with secondary walls were more resistant to depolymerization associated with drying and were resistant to degradation at temperatures below 150°C. Degradation in solvents used to replace the water was greatest for the dioxane, which had the lowest boiling point, and was progressively less as the boiling points increased. The presence of traces of peroxides in the dioxane may have contributed to the degradation. The effect of weathering on cotton fiber is frequently pronounced and certain types of degradation are associated with areas of high light intensity. The initial photochemical oxidation is rapid at first and gradually levels off after satisfying the more easily oxidizable bonds. A Keir boil of 1 hr., which breaks damaged oxygen bonds, had no effect on the D.P. of fiber from unopened bolls that had not been exposed to light, but readily lowered the D.P. of field-exposed cotton. The data show two types of damage, one of which is evident immediately, the other after boiling in alkali. Cotton fiber exposed to sunlight for 90 days showed only slightly lower D.P.'s than fiber exposed for 43 days, which indicates that the rate of depolymerization is rapid at first and then decreases with time. The D.P.'s of the fibers from the major portions of the seeds were fairly uniform and relatively high, whereas those of the fibers from the chalazal caps, where a preponderance of thin-walled fibers are found, were significantly lower. Within the cotton boll the highest D.P.'s were at the bases of the locks, with a progressive decrease from base to tip.
Text. Research J. Aug. 1949 Authors

Decomposition of Jute and Cellulose

Fungal decomposition of jute fibre and cellulose. I. A preliminary survey of commonly occurring species. S. N. Basu. *J. Text. Inst.* **39**, T232–48 (July 1948).

The action of 69 strains of fungi on sized and desized jute, filter paper, and starch was studied. Starchy sizing materials stimulated both growth and staining of the jute fabric by the micro-organisms. 33 strains were found capable of growing on filter paper, causing a loss in weight of from 6% to 76% after 4 weeks of incubation at 30°C. *Penicillium islandicum, Aspergillus fumigatus, Aspergillus terreus, Chaetomium globosum, Chaetomium indicum,* and other species of *Penicillium* represented the most active of the ones tested. **II. The effect of some environmental factors.** Physiological studies were conducted on the decomposition of jute and filter paper by the fungi *Aspergillus fumigatus, Aspergillus terreus, Chaetomium indicum, Paecilomyces varioti, Penicillium citrinum, Penicillium islandicum, Stachybotrys atra,* and *Trichoderma viride.* Generally, jute was more resistant than cotton, which in turn was more resistant than filter paper. Part of the resistance of jute may be due to the natural antibiotic found in the jute extract. The optimum temperature was approximately 30°C for most of the organisms, although *Stachybotrys atra* preferred a lower range, around 25°C. The optimum pH was 5.8. Different responses to nitrogen sources were displayed. In general, peptone and nitrate gave the best growth, while asparagine was used by a few species but not by the others. Ammonium proved the least satisfactory. Growth factors of the vitamin B group—namely, nicotinic acid, riboflavin, thiamin, calcium pantothenate, pyridoxine, and biotin—did not stimulate cellulose decomposition. When grown in mixed cultures, *Aspergillus terreus* was retarded by *Penicillium citrinum* and by *Trichoderma viride. Aspergillus fumigatus* was stimulated by *Paecilomyces varioti* and *Penicillium citrinum,* and *Chaetomium indicum,* by *Paecilomyces varioti.* R. G. H. Siu
Text. Research J. Aug. 1949

Jute Micelles

On the size of micelles in jute fiber of different qualities and of known strength. S. K. Chowdhury and S. C. Sirkar. *Indian J. Phys.* **22**, 39–42 (1948) (through *Chem. Abstr.* **42**, 8887*d* (Nov. 20, 1948)).

The half-widths of reflection from (002), (020), and (120) planes in the x-ray diffraction patterns due to samples of 9 different qualities of jute fiber were determined using blackening-log intensity curves. The values of m_1, m_2, and m_3, which denote the number of times the unit cell is repeated in the micelle along the *a*, *b*, and *c* axes, respectively, were determined in each case by Laue's method. The average strengths of the fibers in samples cut from adjacent portions of the samples used for the x-ray investigation were also measured. It was observed that the value of m_3 is almost constant and is about 9 in the case of 8 of the 9 samples and 7 in the other. The values of m_2 varied from 7 to 12, and seemed to

be proportional to the strength of the fiber. The values of m_1 also varied from 5 to 8, but there seemed to be no correlation between these values and the strength of the fiber.
Text. Research J. Aug. 1949

Structure of Jute

X-ray investigation of the structure of jute. M. K. Sen and H. J. Woods. *Nature* **161**, 768 (May 1948).

The effect of the non-cellulosic constituents of jute on the x-ray structural features of the fiber was discussed. Interferences associated with the lignin and xylan are listed. Lignin was found to modify the response of the fiber to mercerization. J. P. Tordella
Text. Research J. Aug. 1949

Surface of Dyed Nylon

Replica studies of dyed nylon. F. A. Hamm and J. J. Comer. *Anal. Chem.* **20**, 861–70 (Sept. 1948).

Dyed nylon surfaces are examined by the electron microscope using the silica replica technique. The specimens used are thin silica replica holding the original dye crystals which have been "transplanted" from the nylon fiber surface to the silica. This silica replica-dye crystal combination is interpreted as though the original dyed nylon fiber were being examined. The technique probably produces no serious artifacts and the micrographs provide information concerning the size, shape, and distribution of the dye crystals on the original fiber. 15 electron micrographs (7,500 ×) are included. These show nylon dyed with Indanthrene Golden Yellow IGK, Indanthrene Brilliant Pink IR, and Indanthrene Brown IRRD both before and after 2 hrs. steaming at 10 lb. pressure. The striated surface of the nylon is apparent in all micrographs. The steaming results in an increase in the size of the dye crystals in each case. When the steamed dyeings are compared with the original dyeings, each sample shows a loss in tinctorial strength, a loss in fastness to crocking and an increase in fastness to ultraviolet light (carbon-arc Fadeometer). The three dyes on the fiber were identified by their electron diffraction patterns. The method should be useful in identifying a dyestuff on a fiber using only a few mg. of cloth and a few g. of the dyestuff.
A. R. Macormac
Text. Research J. Aug. 1949

Padding

A laboratory study of padding. Part I. Cotton. C. L. Zimmerman and G. L. Royer. TEXTILE RESEARCH JOURNAL **18**, 615–27 (Oct. 1948).

The published literature on fundamental aspects of padding and liquid removal by squeeze rolls in the textile industry is meager and is concerned almost entirely with variations in liquid pick-up. The amount of dye deposited on a fabric during the padding operation depends primarily upon: (1) the concentration of the dye in the pad bath, (2) the percent pick-up, and (3) the ratio of dye-to-water picked up; *i.e.*, the amount of preferential sorption of either dye or water. The first of these variables is easily controlled in the preparation of the padding solution. The present study covers the factors which control the last two variables—*i.e.*, pick-up and preferential sorption. The results for pick-up are expressed as "percent pick-up" and for preferential sorption of dye or water as "dye pick-up factor" (abbreviated D.P.F.). The percent pick-up is defined as the weight of the pick-up multiplied by 100 divided by the weight of the cloth before padding. The D.P.F. is defined as 100 times the ratio obtained by dividing the amount of dye actually on the cloth, as determined by analysis, by the amount of dye that should be on the cloth as calculated from the pick-up and concentration of the pad liquor. Some of the variables in padding found to affect the pick-up when padding cotton cloth are the construction of the cloth, the condition of the cloth, the temperature of the pad bath, the addition of wetting agents, the time of immersion, the speed of the padder, the number of dips and nips, and the pressure (lb. per sq. in.) between the squeeze rolls. The pressure between the squeeze rolls depends upon the total force applied to the rolls, the hardness of the rolls, the size of the rolls, and the thickness of the rubber covering on the rolls. It has been shown through the use of the "dye pick-up factor" that preferential sorption of either dye or water can take place in padding. Factors which have been shown to affect the preferential sorption when padding cotton cloth are the construction and constitution of the cloth, the dye used, the temperature of the pad bath, the addition of assistants to the pad bath, the time of immersion, and the speed of the padder. Authors
Text. Research J. Aug. 1949

Wool Fiber Behavior

The mechanism of set and supercontraction in wool fibers. I. The nature of the chemical reaction which takes place on boiling wool in alkaline buffers. S. Blackburn and H. Lindley. *J. Soc. Dyers and Colourists* **64**, 305–14 (Sept. 1948).

The work of Astbury and Woods (*A*) is reviewed in which it was shown that "set" could be divided into two parts: (1) temporary set, which can be destroyed by the further action of steam on the released fibers, and (2) permanent set, which cannot be destroyed by steam. (*A*) suggested that the temporary set is accompanied by very rapid side-chain hydrolysis and if the fiber were released (by steaming) the long-chain molecules coiled up and the fibers supercontracted. Simultaneously, a linkage rebuilding also occurred which eventually overtook the hydrolytic reaction and set the fiber at some length greater than its original length. Speakman (*B*) proposed that the initial side-chain hydrolysis reaction of (*A*) breaks the cystine disulfide linkages and offered experimental evidence that amino groups are concerned in the linkage-rebuilding reaction. Using the body portion

of a carefully selected 56's New Zealand cross-bred wool, Blackburn and Lindley obtained results generally confirming those of (*A*). They suggest that the most important factor in studying wool under the conditions of (*B*) is the change of temperature and pH undergone by the fiber in the washing process. **II. Effect of esterification of the carboxyl groups of wool on supercontraction in sodium bisulfite and set in aqueous-media.** Wool fibers were first methylated using methyl iodide or methyl bromide, after which supercontraction of treated and untreated fibers was produced in boiling 5% sodium bisulfite solution. The behavior of the methylated fibers differs from that of the corresponding untreated fibers, the percentage set being less for the untreated fibers. The chemical evidence here offers no support of the view that cross-linkages, such as the —S—NH— group, are formed either when wool is boiled in sodium bisulfite or during the setting of strained fibers, further confirming the theory of Astbury and Woods.
Text. Research J. Aug. 1949 C. Amick

Shrinkage Control of Woolens

Function of methylated melamine-formaldehyde resin in controlling shrinkage of woolens. C. R. Stock and D. J. Salley. TEXTILE RESEARCH JOURNAL **19**, 41–3 (Jan. 1949).

The role of melamine resin in controlling felting shrinkage has been investigated by examining the effect of the resin on (*a*) modification of hysteretic response of fibers to stress, (*b*) change in friction of the fiber surface, and (*c*) network formation by bonding of fibers at points of contact. Data fail to support (*a*), give questionable credence to (*b*), but make (*c*) appear most plausible as the controlling mechanism. A quantitative relationship has been derived between shrinkage and enhancement of strength in untwisted threads from treated cloth. Authors
Text. Research J. Aug. 1949

BLEACHING: DYEING: FINISHING

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Nylon Dyeing

The application of chrome dyes to nylon textiles. H. R. Hadfield and D. N. Sharing. *J. Soc. Dyers and Colourists* **64**, 381–6 (Dec. 1948).

Acid dyes on nylon are much faster to wet processing than they are on wool, but the expected improvement of chrome dyes on nylon was not obtained because of incomplete chroming of the dye on the fibers. A method has been evolved which insures complete development of the metal complex within the nylon. This yields dyeings which are capable of withstanding severe washing treatment and in some cases soap and soda boiling, with little or no loss in depth and only slight change in hue. The fastness to light of dyeings prepared from the majority of chrome dyes applied by the newly developed technique is 5–6 in medium depths. Experimental evidence has shown that the behavior of potassium dichromate is different on nylon from that on wool and sufficient experimental evidence has been obtained to provide a general picture of the mechanism governing the application of chrome dyes to nylon. This includes measurement of the absorption of potassium dichromate by undyed nylon and by nylon dyed with chrome dyes. By measurement of the reflectance curve of chrome-dyed nylon the rate of formation of the chromium complex within the nylon has been determined. Evidence has been obtained which suggests that the groups in nylon responsible for the absorption of dye are responsible also for the absorption of dichromate ion. In heavy dyeings, where these groups are practically saturated by the dye, the absorption of dichromate is partly effected by reduction of the dichromate within the nylon by formic acid, a reaction which produces further absorption of dichromate due to the alteration in charge from the dichromate anion to the chromic cation. This reduction mechanism appears to be catalyzed by the dye. Nylon can be readily dyed by a modified top-chrome method wherein 2.5% of sodium thiosulfate is added to the dye bath at the end of the usual chromation period and the dyeing is continued for 30 min. following this addition. This work appears to be predicated upon the thesis that in the application of a chrome dye to wool the chromate or the dichromate ion is first absorbed and then reduced by the wool, after which it is capable of chelating with the dye molecule to produce the chromium complex.
Text. Research J. Aug. 1949 C. Amick

Leveling Agents

Evaluation of leveling agents. D. H. Gunther. South Central Section of the A.A.T.C.C. *Am. Dyestuff Reptr.* **38**, P236–8 (Mar. 7, 1949).

If an agent does more than merely cause better wetting and hence better penetration of dye liquor into the fiber, methods must be devised for evaluating its leveling action. A true leveling agent should slow down the rate of exhaustion so that dye molecules can reach less readily accessible fiber surfaces. The dyer accomplishes this effect by controlling his initial temperatures so that initial dyeing is very slow, adding portions of the dye at intervals, or controlling the movement of the dye liquor with respect to the fiber surface. The action of leveling agents on cotton and wool may be quite different. Since there are no methods for evaluating leveling agents which have universal acceptance, a number of different methods are presented which may be used, and their effects are discussed in some detail. C. A. A.
Text. Research J. Aug. 1949

Size Equipment and Slasher Creels

So you want good cloth. F. D. Hering. *Textile Bull.* **75**, 53, 54, 56, 58 (Feb. 1949).

Care of size preparation equipment to prevent the formation of lumps in the size is discussed. The differ-

ent types of slasher creels and the correct methods of operation are explained. A. L. Landau
Text. Research J. Aug. 1949

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

★

Animalization of Fibers

Animalization of cellulosic fibers. D. Fornelli. *Am. Dyestuff Reptr.* **37**, 853–8, 882 (Dec. 27, 1948).

Cellulose may be animalized by means of: (1) protein substances (frequently casein), which are added to the rayon spinning bath immediately before spinning; (2) non-protein N-containing substances, which include amines, polyamines, amides, polyamides, their reaction products, quaternary ammonium salts, etc.; (3) ethylene amine and its reaction products, particularly those with isocyanates; (4) pheno plastics, amino plastics, synthetic resins derived from cyanamide, and polymerization products, such as the reaction products of amines with polyvinylic alcohols; and (5) mixtures or reaction products of protein substances with N- or S-containing non-protein substances, such as the reaction products obtained from casein with phenyl isocyanate solubilized in aniline. A section is devoted to dyeing and finishing animalized fibers and an excellent, extensive bibliography is attached.
Text. Research J. Aug. 1949 C.A.A.

Orlon Acrylic Fiber

Orlon acrylic fiber—A new synthetic. J. B. Quig. *Papers of AATT* **4**, 61–8 (Mar. 1949).

The characteristics, properties, and probable uses for Orlon, du Pont's newest synthetic, are described and compared with other synthetic fibers. Orlon continuous filament is claimed to be the synthetic most closely resembling silk while Orlon staple surpasses other synthetics in simulating the characteristics of wool. Orlon is superior to nylon in sunlight-resistance, tensile strength, high flex life, and resistance to stretching, acid reaction, insects, fungus, and mildew. Nylon is said to have a greater abrasion-resistance, more resistance to alkalies, and regains a greater percentage of strength in air at high temperatures. Orlon is not a single fiber but is the trade name given to a group of Orlon acrylic fibers.
A. L. Landau
Text. Research J. Aug. 1949

New Comber

Whitin's new model "J" comber. Anon. *Whitin Review* **15**, 1–9 (Nov. 1948).

Presented is a completely new comber that combines high production with superior quality plus parts that are readily accessible, easily serviced, and quickly adjusted.
Text. Research J. Aug. 1949 L. A. Fiori

Picker Beater and Fan Speeds

Picker beater and fan speeds. R. Z. Walker. *Textile Bull.* **75**, 55–6, 58 (Jan. 1949).

A discussion of the proper speeds for picker beaters and fans. Beats per inch given the stock are governed by staple length and the amount of waste present in the stock. The beater speeds should be high enough to obtain satisfactory cleaning without weakening the fibers through excessive beating. A. L. Landau
Text. Research J. Aug. 1949

Roving Frame Roller Weighting

Roller weighting on speed frames. Anon. *Platts Bull.* **6**, 104–6 (Nov.–Dec. 1948).

This new type of saddle weighting system embodies a special arrangement of fixed and sliding fulcrum and pressure points so that these essential points are met: (1) pressures applied are both correct and constant at the points of application to the drafting system irrespective of the relative positions or settings of the rollers; (2) pressure applied or removed is accomplished with a minimum of effort and time; and (3) the system is simple and unobstrusive, maintaining performance over a reasonable working life under average conditions. The principle is described at length and illustrated by both sectional and close-up views.
Text. Research J. Aug. 1949 L. A. Fiori

Woolen Carding

Some factors governing the transfer of material to a worker from a swift. P. P. Townend. *J. Text. Inst.* **39**, T385–94 (Dec. 1948).

A series of experiments has been made to determine the contributions of some of the variables which affect the weight of material transferred from the cylinder to the workers of carding machines. The materials used were chiefly Merino wool or fibers of like diameter. The factors studied were the position of the worker on the cylinder, the relative surface speeds of worker and cylinder, the production of the machine, and the condition and density of the card clothing. Figures are given showing how, and to what degree, these variables affect the weight of material transferred to the worker from the cylinder. Author
Text. Research J. Aug. 1949

Wool Opener Saves Labor Costs

Wool opener saves one-third labor costs. Anon. *Textile Inds.* **113**, 110 (Feb. 1949).

Production of from 4,000 to 8,000 lbs. of wool per hr. is claimed to be obtained on a new opener which may be used to replace feeders, single-cylinder openers, and 2-cylinder openers. The stock is delivered from the machine very evenly sheeted and well cleaned.
A. L. Landau
Text. Research J. Aug. 1949

MISCELLANEOUS

★

Effect of Textiles on the Human Body

Some physiological effects of textiles on the human body. J. Boyd.

J. Text. Inst. **39**, T319–38 (Oct. 1948).

Report of a study conducted for military reasons on the effects of clothing upon the efficiency and comfort of wearers in a hot, humid climate. 3 types of clothing were selected for the study: a British army gabardine and two Japanese army fabrics. All 3 were cotton fabrics, weighing 5.5, 9.75, and 8.0 oz. per sq. yd., respectively. The test consisted of having two subjects wear the clothing and perform work (stepping up and down from a 1-foot stool 12 times each min.) in a controlled room of dry bulb 37.8°C and 76% R.H. The critical values measured were: total weight lost, effective evaporation, maximum rectal temperature of each subject during a test period, and rectal temperature rise (the difference between the subject's rectal temperatures at the end and the beginning of a test period). During the trials it was found that the pulse rate showed so slight a variation that it could not be used as a measure of the heat load imposed by the different suits. With the British gabardine, sweat had dripped to such an extent on the inside of the suits that the subjects' socks were completely saturated and there was free liquid inside the boots. However, both subjects preferred the water-repellent gabardine to either of the Japanese fabrics. The conclusion was that coolness is related directly to the thickness and inversely to the weight, the amount of water absorbed by the suit, and the time required to dry the suit. The gabardine gave the lowest sweating rates and the highest effective evaporation, thereby indicating that it imposed the lowest heat load of the 3 fabrics tested. There are 8 tables and 5 figures presenting the physiological data. A wear test was conducted under combat conditions and again the gabardine was superior. An investigation was also conducted on the effects of various designs of equipment (water bottle, haversack, ammunition, etc.) to be worn with the suits. No significant differences were found between the heat loads imposed by the various types of equipment designs tested. 6 references. J. Tallant

Text. Research J. Aug. 1949

Plastic Laminates

Textile-reinforced plastics. W. J. Brown, *Inst. Plastics Ind., Trans.* 22–39 (Apr. 1947); cf. *C.A.* **42**, 1081g (through *Chem. Abstr.* **43**, 435a (Jan. 10, 1949)).

High strength in laminates prepared with one-step NH_3-catalyzed resin is favored by high tenacity and high elastic modulus of the textile fibers, high tensile strength of the resin, minimum twist in the yarn, minimum crimp in the fabric, maximum resin penetration into the yarn, and high molding pressure. Scouring of the fabric to remove sizing agents greatly increases tensile, shear, and impact strengths of the laminates.

Text. Research J. Aug. 1949

Serviceability

Fabric serviceability—the key to better business. C. W. Dorn. *Am. Dyestuff Reptr.* **38**, 87–8 (Jan. 24, 1949).

Factors that enter into serviceability of merchandise are color-fastness, washability, ability to withstand dry-cleaning, bleaching of colors by chemicals or sunshine, perspiration stains, shrinkage, flammability, water-resistance, and gas fading. In view of the thousands of dollars which are being spent annually in a study of these subjects, consumer education should be given. C.A.A.

Text. Research J. Aug. 1949

New Type Spindle

New Era Spindle in two types. Anon. *The Saco-Lowell Bull.* **20**, 11–24 (Dec. 1948).

An important forward step in spindle making is announced with the introduction of the New Era spindle in 2 types—rubber-mounted and oil-cushioned. Distinguishing features of the former spindle are: (1) plumbing is done accurately and simply while the spindle is motionless; (2) 25–30% saving in power consumption is realized; (3) tape life is increased by at least 300%; (4) relubrication of bearings is necessary about every 18,000–30,000 hrs. The oil-cushioned type possesses somewhat the same advantages, except that it must be plumbed in the conventional manner. The rubber-type spindle consists of 3 sub-assemblies: (1) the steel center shaft with its bearings and accessories, (2) the tube, whorl, and top cap, and (3) the rubber mounting and plumbing device. Each of these assemblies is illustrated and explained at length. In addition, a wooden bobbin and different types of paper tubes specially adapted to this new type of spindle are described and illustrated.

Text. Research J. Aug. 1949 L. A. Fiori

Textile Research, 1948

Textile research achievements in 1948. J. B. Goldberg. *Papers of AATT* **4**, 75–80 (Mar. 1949).

These achievements are classified in 6 groups: (1) fibers and yarns, (2) manufacturing methods and equipment, (3) finished goods, (4) laboratory instruments and techniques, (5) research organization activities, and (6) future developments. Some of the items listed are: under (1), Vicara fiber, Vinyon N, Fiber A, Fiber V or Terylene, and Monocord; under (2), an improved fiber blender, a new vertical cotton opener, a new high-speed comber, a new aid in the winding and twisting of synthetic yarns, a new high-speed drying unit for slashers, and many improvements on spinning and weaving machines; under (3), a new color—Alcian Blue, new anti-static agents for yarns and fabrics, a new resin for imparting stability to cotton, new colored inks suitable for offset printing which are resistant to sunlight fading and severe laundering, and the Tensitrol system for processing all kinds of fabrics at high speeds without tension; under (4), an automatic micrometer adapted for recording yarn diameters continuously, an automatic yarn testing machine which provides a continuous record of breaking strength and elongation, and many other instruments and techniques.

Text. Research J. Aug. 1949 C.A.A.

TEXTILE RESEARCH INSTITUTE, INC.

10 EAST 40TH STREET, NEW YORK 16, N. Y.

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